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DEPARTMENT OF AGRICULTURE,
DIVISION OF CHEMISTRY.
BULLETIN No. 12.

METHODS OF ANALYSIS
OF
COMMERCIAL FERTILIZERS.

PROCEEDINGS
OF THE
THIRD ANNUAL CONVENTION
OF THE
ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS,

AUGUST 26 AND 27, 1886.

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LETTER OF TRANSMITTAL.

UNITED STATES DEPARTMENT OF AGRICULTURE,
DIVISION OF CHEMISTRY,
Washington, D. C., September 9, 1886.

SIR: The benefit conferred on scientific agriculture in the United States by the publication from the Department of the proceedings of the last meeting of the Official Association of Agricultural Chemists has been recognized in every quarter.

The scope of the Association has now been greatly enlarged, in harmony with the views expressed by you in your address published in Bulletin No. 7. The proceedings of the last meeting, held, on your invitation at the Department I now have the honor to place before you for your approval, with the request that, in the interests of agriculture, they be published as a bulletin of the Division of Chemistry.

Very respectfully,

H. W. WILEY,
Chemist.

HON. NORMAN J. COLMAN,
Commissioner of Agriculture.

ANALYSES OF COMMERCIAL FERTILIZERS.

PROCEEDINGS OF THE THIRD ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS AT WASHINGTON, D. C., AUGUST 26 AND 27, 1886.

Edited by CLIFFORD RICHARDSON, *Secretary.*

MORNING SESSION, THURSDAY.

The Association assembled in the rooms of the Chemical Division of the Department of Agriculture at 10 o'clock, Dr. H. W. Wiley, the president, in the chair.

There were present and registered as members of the Association:

Dr. H. W. Wiley, chief chemist, U. S. Department of Agriculture, Washington, D. C., president.

Clifford Richardson, assistant chemist, U. S. Department of Agriculture, Washington, D. C., secretary and treasurer.

W. J. Gascoyne, State chemist, Richmond, Va., of the executive committee.

William C. Stubbs, State chemist, Baton Rouge, Louisiana.

P. E. Chazal, State chemist, Columbia, South Carolina.

John A. Myers, State chemist, Agricultural College, Mississippi.

E. H. Jenkins, chemist, Connecticut Agricultural Experiment Station, New Haven, Conn.

W. W. Cooke, State chemist, Burlington, Vermont.

H. C. White, State chemist, Athens, Georgia.

W. E. Moses, State chemist, Knoxville, Tenn.

M. A. Scovell, director Kentucky Agricultural Experiment Station and State chemist, Lexington.

William McMurtrie, University of Illinois, College of Agriculture, Champaign.

B. B. Ross, assistant to State chemist, Auburn, Alabama.

N. T. Lupton, State chemist, Auburn, Alabama.

G. S. Fellows, State chemist, Agricultural College, Maryland.

There were also present in the convention:

Mr. A. de Ghequier, representing the American Fertilizer Association; Dr. Leffman, of Baltimore; and Messrs. Edgar Richards, A. E.

Knorr, T. C. Trescot, and F. V. Broadbent, assistants in the laboratory of the U. S. Department of Agriculture, who were invited to take part in the proceedings without vote.

In calling the convention to order the president read the following letter from the honorable Commissioner of Agriculture to the executive committee:

UNITED STATES DEPARTMENT OF AGRICULTURE,
Washington, D. C., August 7, 1886.

GENTLEMEN: The results of your deliberations last year, as published in Bulletin No. 7, have proved of great advantage, not only to the chemists engaged in agricultural work throughout the country, but also to the farmers generally.

The existence of such an association as yours is a necessity, and I desire, in every way possible, to promote its efficiency. Permit me, therefore, to extend to you again an invitation to meet at the Department of Agriculture and to offer all the facilities in my power to make your approaching meeting agreeable and successful.

There are still many problems connected with agricultural chemical analysis which demand your serious attention. Permit me also to suggest that it would both be wise and profitable for you to extend the scope of your investigations to other subjects than those immediately connected with the analysis of fertilizers. It seems to me that there is no subject within the scope of agricultural chemical analysis which does not properly fall within the range of your discussions. I am anxious to see all the agricultural chemists in the country participating in your work and contributing to its efficiency.

I hope to be able, as last year, to aid in bringing the results of your deliberations prominently before the people, and promise you every assistance in my power to that end.

Again extending to you the courtesies of the Department, I am
Respectfully,

NORMAN J. COLMAN,
Commissioner of Agriculture.

To the EXECUTIVE COMMITTEE *of the Association of Official Agricultural Chemists.*

Dr. Wiley then addressed the convention as follows:

GENTLEMEN: For the second time under its present organization our Association has met in Washington. It seems proper, in reviewing the work of our society during the past year, to congratulate you on its results. The proceedings of our last meeting, published through the courtesy of the Commissioner of Agriculture as a bulletin of the Division of Chemistry, have had a large circulation and have attracted much attention. It is gratifying to us to know that this bulletin was reproduced in full in the Chemical News, and received favorable mention in many other scientific journals. But it would be idle to think that the work so well begun is already perfect. A first step forward has certainly been taken, but the road before us is still a long one. Were it not so, the necessity of this and future meetings would not exist. But the success which has already attended our efforts has been great enough to warrant further exertion and give hope of greater achievements. It is evident to every one, however, that in our annual meetings, extending over a period of only a few days, little can be done except to bring together results and compare experiences. The patient and laborious

work which alone can give a solid basis for the business of this association must be done in our laboratories and repeated until it gives a sufficient proof of its accuracy. Happily the methods adopted by our committees secure the accomplishment of this work in the most thorough manner, and I doubt whether any other such system of determining the value of any analytical methods is practiced any where else in the world.

In looking over the means used in other countries to secure uniformity in the application of analytical methods we may find many valuable indications to help us in the work which we have undertaken. In France we find an association of chemists devoted to the interests of sugar production and of the manufacture of alcohol. This association has adopted processes of analysis for the examination of fertilizers which they have recommended for use. Already two bulletins have been issued by this body, the second of which is a neat little pamphlet of 43 pages devoted to a discussion of the best methods of estimating phosphoric acid in fertilizers.

The treatment of the subject is in greater detail than the reports of the various committees of this Association which have had the subject in charge, but I doubt whether the practical results obtained have been any greater than those achieved by the labors of our own members who have had this subject under special consideration. Since some of our number may not have seen the results reached by the French committee, I will give a brief summary of them here. The committee in charge of the work consisted of the following eminent agricultural chemists: MM. Dehérain, Joulie, Aubin, and Dupont. Attention is first called to the importance of taking the sample; an importance which is fully recognized by this Association, but which perhaps has not been fully set forth. The French chemists recognize two kinds of samples, commercial and analytical. The commercial sample should be taken from various parts of as large a quantity of the fertilizer as is accessible and by a person neither interested in the purchase nor sale of the substance. Then it is directed to proceed as follows:

If it is fine powder, as ground phosphates in certain manures, it suffices to pass it two or three times through a sieve with very small meshes, 1 millimeter for example, being careful to grind the material each time in order to pulverize the fragments which the sieve may retain. Spread out the whole in a thin layer on a large sheet of paper and take here and there with a spatula small portions which will give a quantity of 20 grams, from which is taken afterwards the samples to be assayed.

If the sample is in more or less large pieces like those of the rock phosphates, or coarsely pulverized like those of guanos containing conglomerated particles, it is necessary first to reduce it to powder by passing it through a mortar or through a small drug mill. Pass it then through a No. 60 sieve and grind again the remainder on the sieve until all passes entirely through. This point is very important, as the particles which resist the pestle frequently have a composition different from those which are broken up at first.

When the products for analysis contain organic materials, such as horn, meat, dried blood, &c., the grinding is often long and difficult, and produces a certain degree of heat which drives off the moisture in such a way that the pulverized prod-

uct is finally drier and consequently richer than the original sample. It is important to note this desiccation, and as the pulverization of a mass is not done without loss, the determination of the total weight of the sample before and after crushing does not give exact results.

It is, therefore, indispensable in this case to determine the moisture before and after the pulverization and to restore by calculations the analytical results obtained in the pulverized samples to that which they would have had in the moist state of the original sample.

To avoid this necessity, as well as those difficulties which would thus result owing to variations in the moisture during transportation, several chemists have thought that the best method of procedure would be to always dry the commercial product before submitting it to analysis, and to give in their reports their value in the dry state accompanied by the determinations of moisture, thus leaving to those interested the trouble of calculating the contents in the normal state, that is to say, the real state in which the merchandise is sold.

In addition to the fact that this method gives rise to numerous chances of error, much of the material undergoes important changes in composition by the mere fact of its drying. It has occasioned, in consequence, the worst frauds.

The retail dealers put these products on sale with a guarantee of their dry state, and a large number of farmers do not perceive the fraud which is hidden under this very innocent expression.

In this way guano containing 25 per cent. of water has been placed on sale as having 12 per cent. of phosphoric acid, when in reality it had only 8 per cent.

Having thus secured as fair a sample as possible, it is recommended, inasmuch as the uranium method is to be used in the determination, to ignite it in order to destroy organic matter. In respect of the method of doing this the French chemists are silent, and it appears from this, as well as many other parts of the report, that they have not studied the publications of our society as carefully as they ought to have done.

The solution of the phosphate is a matter, however, to which they have given much attention, and on this point they make many valuable suggestions.

SOLUTION OF THE MATERIAL.

All phosphates, with the exception of certain aluminum phosphates (amblygonite, for example), easily dissolve in nitric and hydrochloric acids more or less dilute, especially at the boiling point. But the best solvent of calcium phosphates is, undoubtedly, hydrochloric acid, which also dissolves iron and aluminum phosphates which are often found in phosphate of lime.

NITRIC ACID.

In many laboratories nitric acid is preferred, to avoid, as much as possible, the solution of the sesquioxide of iron, which interferes with the determination of phosphoric acid in certain methods of analysis.

Since this is not the case with the citro-uranic method, it is preferable to employ hydrochloric acid, because it completely dissolves the iron, and thus permits one to judge of the completeness of the analysis by the perfect whiteness of the remainder.

PYRITICAL PHOSPHATES.

Certain phosphates, as those of the Ardennes and of the Meuse, contain iron pyrites which hydrochloric acid does not dissolve, and consequently leaves a remainder more or less discolored. It is thus necessary to add nitric acid in the beaker where the

analysis is being made, and prolong the boiling until the pyrites has disappeared, otherwise it will retain a small quantity of phosphoric acid in the state of ferric phosphate.

SULPHURIC ACID.

In Germany many chemists make the analysis of phosphates with dilute sulphuric acid. This method, which certainly gives good results for certain products, presents numerous objections which unfit it for general use. The calcium sulphate which is formed requires prolonged washing, which gives rise to many chances of error.

In the case of aluminum phosphate, containing little or no lime, sulphuric acid may be preferable to hydrochloric and nitric acids, for it attacks amblygonite, which resists the first two acids.

But one seldom meets with such a case, and can always make use of the general method by first fusing the material with the classic mixture of standard carbonate of soda and carbonate of potassium (containing, of course, no phosphate).

In the great majority of cases the solution with hydrochloric acid is made very easily by simply boiling in a beaker and without affecting the separation of the silica. This operation is only necessary after the use of alkaline carbonates or for those substances which contain decomposed silica giving gelatinous silica during the action of the hydrochloric acid.

There are two ways of making the solutions of the sample, which weighs from 1 to 5, or even 10 grams, depending upon the apparent character of the material to be analyzed.

Solution by filtration and washing.—One can work by the ordinary method, consisting of making the solution, filtering, washing the residue on the filter, and mixing the liquids, and making up to a definite volume, and then taking an aliquot part for precipitation.

This method is long, and occasions numerous errors when the insoluble remainder is somewhat voluminous and contains some silica, which fills up the pores of the paper and renders the filtration difficult.

Test for arsenic acid.—When the sample examined is pyritical it often contains arsenic.

Since this is acted on by nitric acid, it can produce arsenic acid. This behaves in all cases as phosphoric acid, and if it is found in the sample it will be precipitated and estimated with the phosphoric acid. It is sufficient, in order to avoid this cause of error, to pass first through the solution a current of sulphurous acid, to carry it then to the boiling point to get rid of the acid, and finally to pass through it a current of hydrogen sulphide, which precipitates the arsenic. Immediately after filtration the series of operations described can be repeated.

Volumetric solution.—We advise the substitution of volumetric solution for solution by filtration and washing. This consists in acting on the sample with the acid in a graduated flask and completing the volume with distilled water. After cooling, filter without washing, and take, by means of a pipette, an aliquot part of the total volume to make the precipitation. Thus the delays and the chances of error of washing upon the filter are avoided. It is true that this method leads to a certain error, due to the volume of the insoluble material which is left; but, as this quantity of insoluble material is generally very small, and as it is always possible to diminish it by increasing the volume of the solution, this cause of error is much less to be feared than those which may be produced in the other method without the knowledge of the operator.

In this last recommendation we have almost the exact method recommended by our former committees. Some objections have been urged against the method on account of the error due to insoluble matter; but this error, in the very nature of the case, is so small as to be negligible when compared with the errors and complexity of manipulation to which any other method of solution is liable.

The French chemists, while admitting the perfect accuracy of the phospho-molybdate method of estimating phosphoric acid, recommend solely the volumetric method first proposed by Joulie. The preliminary precipitation of the ammonio-magnesium phosphate is conducted as follows:

A quantity of the solution representing about .25 grams of the sample, or at most 1.0 gram., if the sample be poor in P_2O_5, is taken. The total quantity of P_2O_5 in the sample taken should be about 50 milligrams. Then 10 cubic centimeters of the citro-magnesian solution is added and a large excess of ammonia. If the proper quantity of citro-magnesian solution has been used, the liquid will now remain perfectly clear.

The citro magnesian solution should be prepared according to the following formula:

	Grams.
Citric acid	400
Carbonate of magnesia (C. P.)	40
Caustic magnesia	20
Distilled water	500

After solution enough ammonia, about 600 cubic centimeters, is added to make the solution strongly alkaline, and the volume is then made up to 1,500 cubic centimeters. If turbid the whole is to be filtered. After the magnesian phosphate is precipitated it should be allowed to stand at rest for some time (2 hours) under cover, to avoid the evaporation of the ammonia. The filtration is made as carefully as possible, washing the precipitate with water containing one-tenth ammonia and not bringing the precipitate onto the filter until near the last. The washing is continued (after all the precipitate is on the filter) until no precipitate is produced by phosphate of soda in the filtrate. The quantity of wash-water should be as small as possible. The phosphate is next dissolved in one-tenth pure nitric acid. The beaker in which the precipitation was made should be rinsed with this acid. The filter-paper should finally be put into the flask receiving the solution. All having been collected in a 150-cubic centimeter flask, marked also at 75 cubic centimeters, one-tenth ammonia is added until a slight permanent precipitate is formed. Two drops of one-tenth nitric acid are then added and the flask placed on a sand bath and raised to the boiling point. Five cubic centimeters acid acetate soda solution is then added, to neutralize the free nitrate acid. The acid acetate of soda solution is made as follows:

		Grams.
Crystallized acetate of soda		100
Strongest acetic acid (glacial)		50
Distilled water, q. s. to make	cubic centimeters..	1,000

The solution of uranium is prepared according to the following formula:

		Grams.
Pure nitrate of uranium		40
Distilled water	cubic centimeters..	800

Dissolve and add ammonia until a slight cloudiness is produced, and then acetic acid to clear the liquid. The volume is then made up to 1,000 cubic centimeters with distilled water.

To obtain perfectly pure uranium nitrate the commercial salt is treated as follows:

The nitrate of uranium often contains phosphate of uranium and ferric nitrate. It is necessary to get rid of the foreign products.

The commercial salt is to be dissolved and precipitated with carbonate of soda which redissolves the oxide of uranium and precipitates the phosphate and oxide of iron.

Saturate the filtered liquor with nitric acid and reprecipitate the oxide of uranium with ammonia. Wash it with distilled water by decantation, redissolve it in nitric acid, as exactly as possible, evaporate and crystallize.

The crystals are taken up by ether, which often leaves a little insoluble residue. Filter and evaporate the ether. The remaining salt is perfectly pure.

The uranium solution prepared as above is much more sensitive than that prepared with the acetate of uranium, as recommended by Neubaur, and this acetate solution should never be used.

The phosphate solution for determining the strength of the uranium solution is prepared from pure acid phosphate of ammonium, a salt which is easily procured. This acid phosphate contains 61.74 per cent. $P_2 O_5$; 8.1 grams pure dry acid ammonium phosphate dissolved to 1,000 cubic centimeters corresponds to .005 $P_2 O_5$ in 1 cubic centimeter.

TITRATION OF THE URANIUM SOLUTION.

The titration of the uranium solution is accomplished as follows:

Standardizing the solution of uranium.—Pour out in a flask (marked at 75 cubic centimeters) 10 cubic centimeters of standard phosphoric acid solution, measured by means of an accurate pipette, add 5 cubic centimeters of the acid acetate of soda and distilled water to make about 30 cubic centimeters and heat to the boiling point. Then titrate by running the solution of uranium into the flask, by means of a graduated burette, stirring after each addition, and testing the liquid, to the tenth, with some drops of ferrocyanide of potassium placed upon a greased dish. As the quantity of solution of uranium ought to be very near 10 cubic centimeters, first add about 9 cubic centimeters without trial. Continue with two or three drops each time, until the test indicates the end of the operation.

When you observe in the last trial a sensible change of color, refill the glass to the mark with boiling distilled water and make a new trial. If in the first part of the operation the point of saturation has not been passed, it is generally necessary to add a drop or two of uranium solution, in order to obtain the characteristic red color, a quantity rendered necessary by the increase of the quantity of the liquor.

This method of procedure permits one to obtain great precision the second time, whereas at first one perceives the point of saturation a little before it has really been obtained.

It is evident that the results obtained are not absolutely exact, for it has been necessary to add not only enough of the uranium solution to exactly precipitate the phosphoric acid, but also enough to give the coloration with the cyanide.

This quantity, however, can be exactly determined, since the same quantity of solution, viz, 75 cubic centimeters, is used for every deter-

mination, by making some blank determinations, *i. e.*, without any phosphates being present. The amount of uranium solution necessary to give the coloration to the ferrocyanide having been determined, it is thereafter accounted for in the titrations. The exact method of making this determination is as follows:

The manipulation is made as in the preceding case: Into a flat-bottomed Bohemian flask of about 150 cubic centimeters capacity, and marked at 75 cubic centimeters; pour by means of a pipette 5 cubic centimeters solution of acetate of soda; add warm distilled water, almost to the mark, and place the flask upon a sand bath heated with gas or otherwise and carry to the boiling point. Take it off the fire, complete the volume to 75 cubic centimeters with a little distilled warm water, and pour in the glass one or two drops of the solution of uranium by means of a graduated burette which has been previously filled to zero. After each drop of the solution of uranium stir and try the liquid upon a drop of ferrocyanide of potassium, as has been previously indicated. For an experienced eye generally four to six drops are necessary in order to obtain the characteristic coloration—*i. e.*, .2 to .3 cubic centimeters. Beginners often use .5 to .6 cubic centimeters and even more.

The only important point is to stop as soon as one certainly sees a red tint, for afterwards the intensity of the color is not increased proportionally to the quantity of the liquor employed.

In the preceding operations three causes of error must be avoided:

The first is the error which may arise from the coloration imparted to the drop of ferrocyanide of potassium from the small quantity of solution removed by the stirring-rod when the eye is not sufficiently experienced.

It is very easy to determine when the end of the reaction has been reached. In order to do this, note the quantity of solution of uranium already employed and add to it four drops; stir and make a new trial upon a drop of ferrocyanide of potassium near the preceding. If the red tint does not clearly appear on taking away the rod it is fair to conclude that it was a mistake, and continue the trial. If, on the contrary, the color appears clearly, the obtained number is taken as correct. It is always well to end the trial by this proof of four supplementary drops, which exaggerate the color and confirm the number found.

The second cause of error, and that which, moreover, is most frequent, consists in overreaching the end of the reaction by operating too quickly. In place of giving a color scarcely perceptible, the trial upon the ferrocyanide gives then a very marked coloration.

In this case the analysis can yet be saved.

There must be in this case a decimal liquor prepared with 100 cubic centimeters of standard phosphoric acid solution diluted to a liter with distilled water. Add to the assay 10 cubic centimeters of this decimal liquor and continue the titration. Account must be taken in the calculation of the quantity of phosphoric acid added.

Finally, the third cause of error is in the froth, which is often produced upon the liquid by stirring. It may retain a portion of the solution of uranium which falls on its surface and prevent its mixture with the remainder of the liquid.

If the glass rod touches this foam charged with uranium, the characteristic color is obtained before the real saturation. It is consequently necessary to avoid, as much as possible the formation of the foam, and above all to have a care to take the drop of the sample, after stirring, only in the middle of the liquid where the foam does not exist."

TITRATION OF THE PHOSPHATE IN THE SAMPLE OF FERTILIZERS.

In this case the quantity of phosphate being unknown, it is necessary to proceed slowly so as not to pass the point of saturation. Since, in this first attempt, a certain error must be introduced by the drops

taken out for trial, the result must not be regarded as final. For the second determination, however, almost the entire quantity of reagent can be added at once and this source of error avoided. All samples should be treated in duplicate. The committee conclude this part of their report with the following observations:

This method of analysis, much longer to describe than to execute, gives results perfectly exact and always concordant, when it is well managed, provided the titrated liquors, upon which reliance is placed, are prepared exactly and frequently verified, as we have indicated.

The standard uranium solution ought to be verified and rectified every three or four days. That of the standard phosphoric acid ought to be tested each time that the temperature of the laboratory undergoes an important change. A solution prepared, for example, in winter, when the temperature of the laboratory is from 15 to 18 degrees, will not be exact in summer, when the temperature is 28 to 30 degrees, at least if care be not taken to cool it before taking the necessary quantities.

The report closes with a very brief description of the methods to be used for estimating soluble and reverted phosphoric acid. These methods, especially for reverted acid, are entirely different from those recommended by our committee. As will be seen, the citrate of ammonium solution is made strongly alkaline, and the precipitation of the phosphoric acid is accomplished with a magnesia mixture in the presence of the citrate. In my opinion the method which is given below is far inferior to the procedure which has been adopted by our committee.

Preparation of the sample for analysis.—The sample sent to an expert chemist is prepared as we have already indicated in the previous pages; that is to say, it is poured in a sieve of which the meshes are 1 millimeter apart, and sifted upon a sheet of white paper. The parts which do not pass through are broken with the hand, or in a mortar, and added to the first portions. The product, well mixed, is introduced into a flask. In this state the mass presents all the homogeneity necessary for analysis. Some fertilizers are sticky, and in this state cannot be sifted. It is necessary in this case to mix them with their own weight of sulphate of lime precipitated and dried at 160°, or sand of Fontainebleau, washed with hydrochloric acid and dried, which divides them perfectly and permits them to be worked through the meshes of the sieve.

Extraction of the products soluble in distilled water.—The products being prepared as has been said, weigh from 1 to 5 grams, and put in the bottom of a glass mortar. Add 20 cubic centimeters of distilled water, mix gently, and after a minute decant the part floating upon the surface into a little funnel furnished with a filter, and placed in a graduated flask of 150 cubic centimeters. This operation is renewed three times, and is terminated by an intimate mixture of the material with the distilled water. When the volume of the filtrate is about 100 cubic centimeters the remainder is poured upon the filter and the washing continued to make the volume to 150 cubic centimeters. Stir to render the solution homogeneous and transfer to a beaker of 300 grams capacity.

Solution of retrograde phosphates with citrate of ammonia.—The filter is taken from the funnel, and is introduced into a graduated flask of 150 cubic centimeters capacity with 60 cubic centimeters of alkaline ammonia citrate prepared in the following way:

Pure citric acid	grams..	400
Ammonia (22°)	cubic centimeters..	500

Pour the ammonia on the citric acid in crystals in a large capsule. The mass is heated and the solution takes place rapidly. When this is complete and cool, pour it into a graduated flask of 1 liter capacity and refill just to the mark with ammonia at 22°. It

is preserved for use in a well-stoppered flask. This solution ought to be strongly alkaline.

The flask in which the filter and citrate of ammonia are put is stoppered and violently shaken to disintegrate the filter and bring into suspension the reverted phosphates. Add about 60 cubic centimeters of distilled water, shake and set aside for twelve hours at least, and twenty-four hours at most. Complete the volume to 150 cubic centimeters with distilled water, and, after mixing, filter.

Precipitation.—Two solutions have now been obtained which can be precipitated together or separately, according to what is desired.

The most ordinary process is to mix quantities of 25, 50, or 100 cubic centimeters, representing .25, .50, or 1 gram of material, according to the presumed value, in a beaker with 10 to 20 cubic centimeters of magnesia solution made as follows:

Carbonate of magnesia	grams..	50
Chloride of ammonium	do....	100
Water	do....	100
Hydrochloric acid	cubic centimeters..	120

Dissolve and add—

Ammonia (22°)	cubic centimeters..	100
Distilled water	Q. S. to make 1 liter.	

Add an excess of ammonia, and leave for twelve hours under a bell glass. The phosphoric acid contained in the liquid separates in a state of ammonio-magnesic phosphate. Gather it upon a little filter, wash it with ammonia water, redissolve and titrate it with the solution of uranium as previously directed.

In respect of this method, employed universally in France and Belgium, Mohr (Chem. Zeit., 1886, No. 44, p. 675) makes the following observations: The direct precipitation by magnesia of phosphoric acid dissolved in citrate of ammonia is employed as a standard method in Belgium and France. As a result of this process it is no unusual thing to find the French analyses falling from 1 to 3 per cent. behind the German. He especially condemns the habit of estimating the soluble and reverted forms of the acid at a single operation. By first removing the soluble and afterward using a much less quantity of the citrate very good results are obtained. The analyses should be made as follows:

Two grams of the superphosphate are rubbed in a mortar with warm water and treated with a proper quantity of acetate of soda. The phosphates of iron and alumina thus produced remain with the residue in filtration. The soluble P_2O_5 is now washed out and the filtrate made up to 200 cubic centimeters. The estimation is then made in an aliquot part, 50 cubic centimeters, in the usual way. The residue is digested with an alkaline solution of citrate of ammonia for an hour at 40° to 50° C., filtered and the P_2O_5 precipitated directly with the magnesia mixture.

In other parts of Europe we may also find associations of official chemists engaged largely in the same kind of work which occupies us here.

Chief among these bodies must be named the English Society of Public Analysts. In this society are discussed all the problems relating to the duties of public analysts, and the methods employed in analytical

work receive a large share of attention. In illustration of this I may call attention to the discussion of the method of milk analysis, which has occupied so large an amount of the time of the society during the past two years. As a result of this work the old methods of estimating fat in milk have been entirely superseded. The society has formally adopted the principle of Adams, which is based on a preliminary absorption of the milk by bibulous paper, and after drying rolling the paper into a coil suitable for insertion in a siphon extractor. Blotting-paper was first proposed by Adams for this purpose, but Johnstone subsequently recommended the use of thick blotting-paper, and this seems to be as successful as the first method. I have had the method partially tried in the laboratory here and with pleasing results. The filtering paper, previously thoroughly extracted with ether, is cut into strips 2½ inches wide and 2 feet long. 5 cubic centimeters of milk from a weighing bottle are dropped along the central portion of this slip of paper, being careful to leave the ends and, if possible, a narrow margin along the sides, dry. The paper is then hung up over a sand bath or other hot place, where it becomes entirely dry in a very few minutes. It is then rolled into a coil and placed in a syphon extractor.

English analysts have found that this method gives fully .2 per cent. more fat in an ordinarily rich milk than in any of the old processes of evaporation of the milk with sand or gypsum.

I only mention the above to give an idea of the kind of work which is accomplished by this society, and which is so entirely in the line of our own investigations.

In Germany there are numerous societies having in general the same object in view. One of these in Bavaria has lately issued two valuable bulletins which show the general character of the work done. This society had various committees appointed for the purpose of considering different subjects. For instance, there were committees for the investigation of the methods of analysis of milk, beer, wine, drinking-water, flour and bread, butter and butter substitutes, tea, coffee, and chocolate productions, &c. All of these committees made reports at the annual meetings, and these with the discussions thereon were printed in the bulletins. In the matter of milk analysis to compare the work of the Bavarian society with that of the English, the committee adopted a report which covers 114 closely printed pages. All the usual methods of analysis are carefully discussed, and the preference given finally to Soxhlet's areometric method. At the time the report was made, however, it is necessary to state that the committee had not seen the recent work of the English society to which I have alluded, and therefore made no recommendation in respect of the tables which are used for the computation of Soxhlet's method, and which are based on the old methods of the gravimetric determination of fat. It is evident, however, that if the results obtained by the English analysts should prove

to be true it will be necessary to make a careful revision of this table in order to reach accurate results by the Soxhlet method.

Having thus briefly called attention to some of the work which is carried on by societies in other parts of the world, having in general the same objects in view as our own, it may be proper to remind you that the scope of our own society appears somewhat limited as compared with those mentioned. It seems to me that our numbers might be materially increased and the good results of our meetings widely extended if our investigations were made to include a wider range of subjects. Every problem connected with chemical agricultural analysis falling within the range of the studies of any one connected officially with the agricultural interests of the country seems to me to be a proper theme for our discussion here. Thus all the problems relating to the adulteration of food, the general method of agricultural analysis, and all other matters which concern, in common, the analyst and the public, could be included among the duties and privileges of this body. In this way large numbers of chemists in official or semi-official life who do not feel that they are properly candidates for membership in our body could be added to our numbers. In consideration of these facts I suggest whether it might not be advisable to appoint a committee to consider the propriety of revising that clause of our constitution which limits our membership and the scope of our investigations.

On the conclusion of the president's address it was moved and carried that the order of business adopted for the previous meeting be continued for the present, as follows:

(1) Reports of committees: (*a*) on Phosphoric Acid; (*b*) on Potash; (*c*) on Nitrogen—the reports to be received and discussed in this order.

(2) Determining methods to be used during the next year, in the same order.

(3) Election of officers.

(4) Miscellaneous business.

In accordance therewith the report of the Committee on Phosphoric Acid was presented by the chairman, Dr. H. C. White, of Georgia, as follows:

REPORT OF THE COMMITTEE ON PHOSPHORIC ACID.

The Committee on Phosphoric Acid respectfully submits the following report:

Adopting the plan followed by the committee last year, the report presents, (1) brief notices of new analytical methods for determining P_2O_5 proposed during the year; (2) results of co-operative work done by the committee and members of the Association; (3) recommendations for the next year.

The publication since our last meeting of the second edition (1886) of Crookes' Select Methods in Chemical Analysis has presented in convenient form outlines of nearly all methods for determining P_2O_5 worthy of note that have been suggested to the beginning of the current year. In addition we note as follows: H. Joulie (Ann. Agronom., *IX*, 97–129; Abstract Chem. News, *LII*, 85), republishes his "citro-uranic" method for estimating P_2O_5 in manures by dissolving in HCl, precipitating as magne-

sium-ammonium phosphate in presence of excess of citric acid, dissolving the precipitate in dilute HNO_3 with addition of sodic acetate, and titrating with uranic nitrate. He compares unfavorably the ordinary nitro-molybdate method. The essential points in the process, with critical remarks, are given in Crookes' "Select Methods, Second Edition, 504–5, and in more detail in Chemical News Abstract (*LII*, 85). J. M. H. Munro adds to the Abstract in Chemical News some examples of tests of Joulie's method, indorsing it if carried out exactly as recommended.

M. Meinecke (Repertorium Anal. Chem., *V*, 153, Chemical News, *LIII*, 53) proposes to determine P_2O_5 by direct weighing of the precipitate of ammonium phosphomolybdate after heating for fifteen minutes at a temperature of 400° to 500° C. The composition of the black compound thus obtained is said to be constant, containing 4.018 per cent. P_2O_5. Certain precautions in precipitating with molybdate solution, washing and drying, are pointed out as necessary.

Tests of this method, made in the laboratory of the chairman of the committee, have given unsatisfactory results.

E. Aubin (Compt. Rend. *C*, 1595–6; Jour. Chem. Soc. Abstract, *XLVIII*, 1093) avoids the precipitation of silica, lime, magnesia, fluorine, &c., and determines P_2O_5 by immediate precipitation with magnesia mixture as follows: The sample is dissolved in boiling HCl with addition of a saturated solution of sodic acetate in acetic acid, solid ammonic oxalate being added during the boiling. The precipitate is allowed to settle, filtered, and well washed. Excess of ammonia and solution of ammonic citrate is added to the filtrate, which is then precipitated in the usual manner with magnesia mixture.

The committee calls special attention to the very interesting and valuable paper of Dr. Paul Wagner (Chemiker Zeitung X, 13; translated in Chem. News, *LIII*, 133–136). Dr. Wagner has attempted by crop experiments to establish the relative manurial value of the "water insoluble" P_2O_5 in different classes of phosphates, compared with that of the "water-soluble," and to devise an analytical method for the determination of what he calls "soluble" P_2O_5 (corresponding to what we designate "available") in either a superphosphate or a phosphate containing no "water-soluble" P_2O_5, which method shall yield results corresponding to the proportionate manurial values.

The method proposed is as follows: 5 grams of the sample are rubbed up with some dilute citrate solution poured into a half-liter flask, made up to the mark with dilute citrate solution, allowed to stand about 18 hours at the temperature of the room (13° to 18° C.), with frequent shaking, and then filtered. Fifty cubic centimeters of the filtrate are taken and the P_2O_5 determined in usual manner by molybdate and magnesia mixture. The citrate solutions are made as follows:

(1) Concentrated citrate solution. 150 grams of citric acid are placed in a liter-flask, dissolved in water and neutralized with ammonia. To the neutralized solution 10 grams of citric acid are added, and the solution made up to the mark with water.

(2) Dilute citrate solution. 1 volume of concentrated citrate solution is mixed with 4 volumes of water.

The other reagents are essentially the same as those now used by this Association.

At a meeting of the manure manufacturers of South Germany and representatives of the experiment stations of Bonn, Darmstadt, Marburg, Spier, and Wiesbaden, held at Mayence November 30, 1885, this method for the analysis of all superphosphates was unanimously agreed upon to be used from January, 1886, and the term "soluble" was adopted to designate the P_2O_5 extracted by the process indicated.

In view of the possible importance of the method suggested should it be generally adopted by continental chemists, the chairman of the committee has made a few experiments to test the results given.

The samples of superphosphates A, B, and C, distributed to members of the Association, as hereinafter described, were used in the experiments.

(1) Five grams of the superphosphate were treated directly by Wagner's method, with the following results:

	A.	B.	C.
"Soluble" (Wagner)	8.35	12.10	9.95
	8.37	12.15	10.01
	8.40	12.14	9.96
Mean	8.37	12.13	9.97

(2) Five grams were first exhausted with pure water, as in our ordinary method of determining "water-soluble," and the residue was then treated by Wagner's method.

	A.	B.	C.
Water-soluble	7.40	11.50	9.15
Obtained from residue	2.25	1.52	1.75
	2.20	1.50	1.68
Mean	2.23	1.51	1.71
"Soluble" (Wagner)	9.63	13.01	10.86

It is very evident that Wagner's method gives much lower percentages of what we are accustomed to call "available" P_2O_5 than the method now in use by us. The introduction of this method into our work would clearly be attended with many embarrassments; and yet, if Dr. Wagner's crop experiments and his conclusions are to be relied on, it is a question if his analytical method is not a proper one to employ in the official work of experiment stations and State officers. It will be remembered that considerations of a similar character led to the adoption by this Association of the temperature of 65° C. for digestion of the citrate solution in determination of "insoluble" P_2O_5.

II. Owing to absorbing engagements of the chairman the committee was somewhat tardy this year in the distribution of samples for co-operative work. Finally, on June 12, three samples of superphosphates were sent to each member of the Association and to others, marked as follows:

A.—Acid Phosphate, South Carolina.

B.—Acid Phosphate, Navassa.

C.—Acid Phosphate, South Carolina and Navassa mixed.

These materials had been specially furnished by courteous manufacturers and were carefully sifted and mixed, so as to secure uniformity. In the circular letter accompanying the samples request was made that the determinations be made within the week beginning Monday, June 28.

Returns have been received from eleven laboratories, covering the work of fifteen analysts. The results in full are here given.

RESULTS OF PHOSPHORIC ACID DETERMINATIONS.

Analyst.	Moisture.	Soluble.	Reverted.	Insoluble.	Available.	Total.
Sample A.						
A	7.15	7.16	4.29	7.61	11.45	19.06
B	7.02	6.63	5.00	7.31	11.63	18.94
C	7.05	7.01	5.33	7.04	12.34	19.38
D		6.98	5.09	7.48	11.07	18.55
E	6.27	7.00	5.05	7.01	12.06	19.06
F	6.27	6.99	5.01	7.04	12.00	19 03
G	7.14	7.01	3.97	7.71	10.98	18.69
H		7.15	3.77	7.77	10.92	18.69
I	6.88	7.05	4.69	7.35	11.74	19.09
K	7.22	7.04	5.24	6.75	12.28	19.03
L	7.17	7.46	5.06	6.42	12.52	18.94
M	8.70	7.12	3.76	8.04	10.88	18.92
N		6.90	4.29	7.87	11.19	19.06
O	7.25	7.40	5.16	6.40	12.56	18.96
P	7.20	7.32	5.09	6.45	12.41	18.86
Sample B.						
A	9.65	11.13	4.08	3.85	15.21	19.06
B	10.06	10.11	5.06	3.41	15.17	18.58
C	10.03	10.72	4.82	3.58	15.54	19.12
D		10.03	4.39	3.87	14.42	18.29
E	9.17	10.51				18.47
F	9.17	10.54	4.26	3.58	14.80	18.38
G	10.14	11.30	3.46	3.56	14.75	18.31
H		11.23	3.23	3.84	14.45	18.29
I	10.16	11.05	4.29	3.49	15.34	18.83
K	10.15	11.39	3.51	3.80	14 90	18.70
L	10.11	11.55	3.88	3.40	15.43	18.43
M	11.87	11.24	3.26	3.82	14.50	18.32
N		10.54	4.12	3.91	14.66	18.56
O	10.20	11.50	3.85	3.10	15.35	18.45
P	10.20	11.58	3.76	3.17	15.34	18.51
Sample C.						
A	10.35	9.08	4.15	5.13	13.23	18.30
B	10.30	8.67	4.27	4.83	12.94	17.77
C	10.40	8.99	4.62	4.72	13 61	18.33
D		8.68	3.50	5.40	12.18	17.58
E	9 62	8.70	4.17	4.91	12.87	17.88
F	9.62	8.75	4.23	4.68	12.98	17.66
G	10.15	8.84	3.44	5.23	12.28	17.51
H		8.88	3.42	5.20	12.29	17.49
I	10.56	8.93	4.23	4.78	13.16	17.94
K	10.45	9.21	3.96	4.83	13.17	18.00
L	10.63	9.25	5.10	3.38	14 35	17.73
M	12.20	9.02	2.94	5.56	11 96	17.52
N		8.76	3.27	5.77	12.03	17.79
O	10.50	9.15	5.20	3.40	14.35	17.75
P	10.61	9.23	5.09	3.43	14.32	17.75

Averages.

	Number of determinations.	Mean.	Highest.	Lowest.
Sample A.				
Moisture	11	7.18	8.70	6.27
Soluble	15	7.08	7.46	6.63
Insoluble	15	7.55	8.04	6.40
Total	15	18.95	19.38	18.55
Sample B.				
Moisture	11	10.16	11.87	9.17
Soluble	15	10.96	11.58	10.03
Insoluble	14	3.57	3.91	3.00
Total	15	18.55	19.12	18.29
Sample C.				
Moisture	11	10.52	12.20	9.62
Soluble	15	8.94	9.25	8.67
Insoluble	15	4.75	5.77	3.38
Total	15	17.80	18.33	17.51

As further illustrating the differences in the results, the following table is presented:

	Percentage of results differing from mean not more than—						
	0.3 per cent.	0.5 per cent.	0.7 per cent.	1.0 per cent.	1.5 per cent.	1.3 per cent.	1.7 per cent.
Sample A.							
Moisture	81	81	81	92	92	100	.....
Soluble	80	100	.....	.....	.....	.....	.....
Insoluble	40	54	74	80	100	.....	.....
Total	87	100	.....	.....	.....	.....	.....
Sample B.							
Moisture	73	73	82	92	92	92	100
Soluble	34	66	87	100	.....	.....	.....
Insoluble	72	93	100	.....	.....	.....	.....
Total	87	87	100	.....	.....	.....	.....
Sample C.							
Moisture	73	82	82	92	92	92	100
Soluble	94	100	.....	.....	.....	.....	.....
Insoluble	40	60	66	74	94	100	.....
Total	80	94	100	.....	.....	.....	.....
All determinations (not including moisture) taken together	68	84	92	95	99	100	.....
P_2O_5 determinations excluding insoluble	77	91	98	100	.....	.....	.....

In connection with these results the following points may be noticed:

1. The determinations of moisture, while somewhat better than those reported last year, are not as uniform, by any means, as could be desired.

2. The determinations of total P_2O_5 on the whole are satisfactory. The extreme variations between the highest and lowest results are for A, 0.83 per cent.; for B, 0.83 per cent.; for C, 0.82 per cent. Of the results upon A, 0.87 per cent. differed from the average not more than 0.3 per cent.; of B, 0.87 per cent., and of C, 0.80 per cent.

In order to determine as accurately as possible the content of total P_2O_5 in the samples numerous special determinations were made by the chairman of the committee. Reagents and manipulation were proved satisfactory by determinations made upon solutions of C. P. hydro-disodic phosphate and of microcosmic salt, recrystallized, the strengths of which were proven by evaporation and ignition and by precipitation with magnesia mixture. A series of molybdate determinations gave a mean error of less than 0.15 per cent.

(1) 0.5 gram of sample was digested with 20 cubic centimeters concentrated HCl, diluted and filtered.

(2) 0.5 gram was covered with 5 cubic centimeters saturated solution of $Mg(NO_3)_2$, evaporated, ignited, and dissolved as above.

The following results were obtained:

	A.	B.	C.
1	19.00	18.48	17.71
	18.94	18.60	17.80
	18.96	18.54	17.76
2	18.89	18.50	17.70
	18.97	18.61	17.81
	18.92	18.49	17.72
Average	18.94	18.53	17.75
Average from Association chemists	18.95	18.55	17.80

3. The determinations of soluble P_2O_5 are fairly satisfactory. The greatest extreme variation is 1.55 per cent. in the case of sample B, and results upon this sample (which

contains the largest percentage of soluble) are much more discordant than for the others. Certain experiments were, therefore, made upon this sample.

1. 2 grms. were placed upon a dry filter and water added slowly and in small quantities until the filtrate measured 200 cubic centimeters; 2. 2 grms. treated as above until the filtrate measured 250 cubic centimeters; 3. 2 grms. treated as above until the filtrate measured 300 cubic centimeters; 4. 2 grms. treated as above until the filtrate measured 500 cubic centimeters; 5. 2 grms. were placed in a beaker, about 150 cubic centimeters of water added, stirred and rubbed with a stirring rod, filtered and washed until filtrate measured 300 cubic centimeters.

An aliquot was taken for determination in each case, after thorough shaking of the fluid and a small quantity of HCl added to clear up any possible cloudiness.

The results were as follows:

	1.	2.	3.	4.	5.
Sample B, soluble P_2O_5	10.52	11.35	11.56	11.55	10.21
	10.53	11.27	11.44	11.51	10.16
Mean	10.58	11.31	11.50	11.53	10.18

These results indicate the necessity for slow washing with small quantities of water and for continuing the washing to the proper point. Acid reaction in the last drop of the filtrate was barely discernible in 2, after 250 cubic centimeters of water had been used, and yet 50 cubic centimeters more extracted nearly 0.20 per cent. soluble P_2O_5.

4. The "insoluble" determinations are extremely unsatisfactory. The extreme variations are, for A, 1.64 per cent; for B, 0.91 per cent.; and for C, 2.39 per cent. As upon this determination depends the estimation of the "available" P_2O_5, its importance is obvious. These discrepancies cannot be due to differences in the samples.

The close agreement in total P_2O_5 evidences strong probability that the samples were uniform. The same fact indicates that there is no fault in the method used for determining the P_2O_5 (molybdate and magnesia mixture). The difficulty is evidently in the character and manipulation of the citrate solution.

The committee is at a loss to know what to suggest in this connection, except it be to reiterate the cautions hitherto urged as to the strict neutrality and specific gravity of the citrate solution and careful attention to the specified temperature.

III. The committee recommends the continuance of the present general method for the determination of phosphoric acid, subject to such modifications in minor details as the Association may determine.

Respectfully submitted.

H. C. WHITE, *Chairman.*
E. H. JENKINS.
W. C. STUBBS.

Upon the conclusion of the reading of the committee's report the president called for the presentation of papers upon the subject.

In response, Mr. CLIFFORD RICHARDSON read the following:

ON THE PROPER INDICATOR FOR DETECTION OF NEUTRALITY IN AMMONIUM CITRATE SOLUTION FOR P_2O_5 DETERMINATIONS.

I have been led to suspect that the cause of some of the discrepancies between analysts in the determination of citrate soluble phosphoric acid lay, at least in a minor degree, in the character of the citrate solution.

The specific gravity is, for instance, to be 1.09, but no temperature is given. Between winter and summer temperatures this might be of some importance.

Neutrality, however, is the point which I think is difficult of obtaining with certainty and uniformity by all chemists.

The question of indicators has been much discussed during the past year, and the principal references I have collected as an appendix to this paper.

The results obtained by the investigators referred to have led me to examine especially the relation of various indicators to citric acid and ammonia.

Solutions were prepared of standard strength, fifth normal, of citric acid and ammonia and observations made of how closely the various reagents indicated neutrality.

The results, together with a comparison of the use of soda in place of ammonia, and oxalic acid in place of citric, are presented in the following table:

Experiments with indicators.

Acid, N/5	C. C. taken.	Alkali, N/5	C. C. taken.	Indicator.	Remarks.
Oxalic	10.0	Soda	10.05	Coralline	Sharp.
	10.0	do	10.05	do	Do.
	10.0	Ammonia	10.0	do	Do.
	10.0	do	9.9	do	Do.
Citric	10.0	Soda	10.1	do	Not as sharp.
	10.0	do	10.0+	do	Do.
	10.0	Ammonia	10.0	do	Less sharp.
	10.0	do	9.9	do	Do.
Oxalic	10.0	Soda	10.05	Lacmoid	End not sharp.
	10.0	do	10.1	do	Do.
	10.0	Ammonia	9.95	do	Do.
	10.0	do	9.9	do	Do.
Citric	10.0	Soda	9.9	do	Indistinct.
	10.0	do	10.3	do	Do.
	10.0	Ammonia	10.0?	do	Uncertain.
	10.0	do		do	Do.
Oxalic	10.0	Soda	10.7	Poirrier's blue	Not sharp.
	10.0	do	10.8	do	Indistinct.
	10.0	Ammonia	12.3	do	Do.
	10.0	do	(?)	do	Do.
Citric	10.0	Soda	10.7	do	Not sharp.
	10.0	do	10.7	do	Do.
	10.0	Ammonia	11.8	do	Indistinct.
	10.0	do	14.0	do	Do.
Oxalic	10.0	Soda	10.5	Methyl orange	Sharp.
	10.0	do	10.6	do	Do.
	10.0	Ammonia		do	Not changed.
	10.0	do		do	Do.
Citric	10.0	Soda	10.6	do	Sharp.
	10.0	Ammonia		do	Not changed.
Oxalic	10.0	Soda	9.6	Cochineal	Sharp.
	10.0	Ammonia	9.6	do	Less sharp, but distinct.
Citric	10.0	Soda	7.0	do	Do.
	10.0	Ammonia	6.3	do	Do.
Oxalic	20.0	Soda	10.1	Phenolphtalein	Sharp.
	10.0	Ammonia	10.1	do	Not so sharp.
Citric	10.0	Soda	10.3	do	Sharp.
	10.0	Ammonia	10.6	do	Not sharp.
Oxalic	10.0	Soda	20.0	Litmus paper	Sharp, but changes.
	10.0	do	10.05	do	Reddens on drying.
	10.0	Ammonia	10.0	do	Paper in liquid.
	10.0	do	10.2	do	Drop method.
Citric	10.0	Soda	10.3	do	Do.
	10.0	do	10.0	do	Paper in liquid.
	10.0	Ammonia	10.0	do	Do.
	10.0	do	10.3	do	Drop method.
Oxalic	10.0	Soda	10.0	do	Paper in liquid.

With coralline in a saturated alcoholic solution, 5 drops used in titration gave a sharp change from yellow to pink.

With citric acid and ammonia the indication was less marked than with soda, but easily distinguished.

Thomsen says he found rosolic acid, the coloring matter of coralline, incompatible with ammonia and citric acid. This is not entirely true, and coralline may be depended on for such indications as it furnishes with enough distinctness to read.

With lacmoid the change from red-brown in acid solution is gradual to a violet

tint, but is easily followed to a standard color except with ammonia, where the change is too gradual.

Poirrier's blue C 4 B in a similar way is easily followed, except with ammonia, from blue to a reddish tinge.

Methyl orange, as described by others, could not apparently be obtained in this country. That used was marked Poirrier's Orange No. 3, and was from four sources. It was turned from yellow to red-brown by soda, giving a distinct indication, but was not affected by ammonia. The point indicated with oxalic and citric acids and soda was not, however, neutrality.

Cochineal, in experienced hands, is a fine indicator, but requires a practiced eye, and is of no value with organic acids and ammonia for determinations of absolute neutrality.

Phenolphtalein is excluded from consideration by its behavior with ammonia.

Litmus paper, as usually prepared, is not reliable. When made by the directions of Sutton* it is very sensitive, and when immersed in the liquid to be tested gives valuable indication. If a drop of citrate of ammonia is allowed to evaporate on the paper it is reddened, however, so that this method of testing cannot be employed.

Several other indicators have been mentioned frequently of late, such as phenacetoline and gallein. These it was not in my power to investigate.

From the results I was led to conclude that coralline and litmus paper, carefully prepared by the method given by Sutton, and immersed in the citrate solution, are the only indicators available for determining neutrality of citrate of ammonia, with a decided preference for coralline. Litmus is not as delicate, and when used by the drop method in the air is quite unreliable.

I would recommend that coralline be prescribed by the committee as the required indicator, and that the specific gravity of the citrate be determined at 20° C.

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Mr. EDGAR RICHARDS then presented a paper entitled—

NOTES ON A COMPARISON OF THE OFFICIAL AND NITRIC ACID METHODS FOR PHOSPHORIC ACID DETERMINATION.

A discussion having arisen in the laboratory of this Department in regard to the use of nitric acid as a solvent in the determination of total phosphoric acid in fertilizers, I undertook some comparative experiments to determine the following points:

Whether there was any difference in the results obtained between the two methods; and

Whether previous ignition with or without the addition of magnesium nitrate was necessary.

Nine samples of fertilizers were taken, eight of which were received from the committees of this Association, viz, Nos. 1–5, the samples sent out in 1885 by the potash committee, and Nos. 7–9, those received this year from the phosphoric acid committee. No. 6, the remaining sample, was the one that occasioned the discussion.

The following tables give the method of analysis followed and the results obtained:

TABLE I.

Official method.		Nitric acid method.	
1.	2.	3.	4.
Take 2 grams.	Take 2 grams.	Take 2 grams.	Take 2 grams.
Add 5 cubic centimeters $Mg(No_3)_2$ and dry.			
Ignite.	Ignite.	Ignite.	
Heat with 15 cubic centimeters HCl.		Boil with 10 cubic centimeters HNO_3	
Filter and dilute to 200 cubic centimeters.		Filter and dilute to 200 cubic centimeters.	
Take 25 to 50 cubic centimeters in duplicate.		Take 25 to 50 cubic centimeters in duplicate.	
Neutralize with NH_4OH.		Neutralize with NH_4OH.	
Add 15 grams Am. nitrate.		Add 50 to 100 cubic centimeters Am. molyb sol. (*)	
+50 to 100 cubic centimeters, Am. molyb. sol(*).		+10 cubic centimeters acid Am. nitrate sol. (†)	
Heat for 1 hour at 60°C. with occasional stirring.		Heat for 1 hour at 80°C. with frequent stirring.	
Filter and wash with dilute Am. nitrate sol.		Filter and wash with dilute acid Am. Nitrate sol. (1 to 1).	
Dissolve with NH_4OH.		Dissolve NH_4OH dilute.	
Neutralize with HCl. Cool.		Neutralize with HCl. Cool.	
Add 5 to 10 cubic centimeters Mg. mixture, and 30 cubic centimeters dilute NH_4OH.		Add 5 to 10 cubic centimeters Mg. mixture (*). and 5 cubic centimeters NH_4OH.	
Let stand 1 hour		Let stand 1 hour.	
Filter, wash, ignite, and weigh $Mg_2P_2O_7$.		Filter, wash, ignite and weigh $Mg_2P_2O_7$	

* Fresenius formula.

† { 650 cubic centimeters HNO_3 sp. gr. 1. 2.
70 cubic centimeters NH_4OH, strong.
330 cubic centimeters. Water.

TABLE II.—*Comparison of official and nitric acid methods for P_2O_5.*

	(1) Low-grade superphosphate.	(2) High-grade phosphate.	(3) Tankage.	(4) Tobacco stems and ground bone.	(5) Ground bone.
(1) Official method	9.776	11.324	12.079	11.567	20.319
	9.750	11.337	12.027	11.464	20.268
(2) Official method, except treatment with Mg $(NO_3)_2$	9.724	11.413	12.232	11.388	20.216
	9.801	11.490	12.283	11.362	20.216
(3) HNO_3 method	9.801	11.439	12.233	11.618	20.396
	9.776	11.490	12.207	11.567	20.421
(4) HNO_3 method, except ignition	9.827	11.464	12.156	11.362	20.268
	9.852	11.516	12.104	11.336	20.268
Highest	9.852	11.516	12.283	11.618	20.421
Lowest	9.724	11.324	12.027	11.336	20.216
Difference	.128	.192	.256	.282	.205

	(6) Tankage.	(7) South Carolina rock.		(8) Navassa.		(9) South Carolina and Navassa mixed.	
(1) Official method	9.007	19.103	19.039	18.489	18.361	17.875	17.785
	8.982	19.076	19.026	18.451	18.399	17.875	17.530
(2) Official method, except treatment with Mg $(NO_3)_2$	8.879	18.758		18.604		17.734	
	8.879	18.860		18.553		17.713	
(3) HNO_3 method	8.995	17.313	17.043	18.374*	17.657	16.897	16.828*
	8.995	17.350*	17.018	18.220*	18.092	16.813	16.851*
(4) HNO_3 method, except ignition	8.956	19.090		18.886		18.349	
	8.892	19.244		18.962		18.297	
Highest	9.007	19.244	19.244	18.962	18.962	18.349	18.349
Lowest	8.879	17.018	18.758	17.657	18.361	16.813	17.530
Difference	.128	2.226	.486	1.305	.601	1.536	.819

The analyses show that the treatment of magnesium nitrate may be omitted, as it does not affect the results one way or the other; and the danger of losing an analysis by its spattering on ignition is avoided and time saved in getting a solution. Mere ignition at a low heat is enough to destroy all organic matter.

The nitric acid method without previous ignition of the fertilizer gives results that agree very closely with the official method, and though the solution may be highly colored yet the organic matter is sufficiently oxidized to be harmless in the analysis. The solution is obtained in a very short time and this method is much preferable for quick and accurate work.

When the fertilizer contains compounds of iron ignition renders these insoluble in nitric acid, and in such cases the destruction of the organic matter may be omitted, or the ignited substance may first be moistened with 2 cubic centimeters hydrochloric acid before the addition of the nitric acid. In the results of Nos. 7–9, marked with an asterisk, hydrochloric acid was used, but in no other cases. In the case of South Carolina rock, however, this method of procedure did not give satisfactory results, as the table of analyses shows, but where the previous ignition was omitted the results were very good.

Instead of taking the trouble in the official method to weigh out 15 grams of ammonic nitrate each time I make a concentrated solution of the salt, so that 10 cubic centimeters of it are equivalent to the 15 grams.

Dr. GASCOYNE then read the following experiments designed to compare the official method for determining total phosphoric acid by ignition with magnesium nitrate with that of dissolving in nitric acid without previous ignition:

Samples of commercial fertilizers containing large percentages of organic matter derived from fish scraps, tankage, azolin, &c., were used in the experiments.

In the nitric-acid method 2 grams of the fertilizer were dissolved in 50 cubic centimeters nitric acid and a few drops of hydrochloric acid, the solution made up to 1 liter, and an aliquot taken representing ½ gram of the material.

	Official method.	Nitric acid method.
No. 1, commercial fertilizer	11.64	11.66
No. 2, commercial fertilizer	10.15	10.12
No. 3, commercial fertilizer	12.01	12.08
No. 4, commercial fertilizer	11.05	11.01
No. 5, commercial fertilizer	12.86	12.77
No. 6, commercial fertilizer	11.90	11.93
No. 7, tankage	13.65	13.68
No. 8, bone meal	23.27	23.26
No. 9, fish scrap	7.17	7.22
No. 10, tankage	9.19	9.16

It will be seen that the nitric-acid method is quite as satisfactory as the official.

The secretary then read, by consent of the Association, a paper contributed by Dr. William Frear and H. B. McDonnell, of the State College of Pennsylvania, entitled—

A COMPARISON OF WAGNER'S AND THE PRESENT ASSOCIATION METHODS FOR THE DETERMINATION OF AVAILABLE PHOSPHORIC ACID.

Some time ago the fertilizer control stations of Southern Germany adopted for their official method for the determination of available phosphoric acid, that employing an acid solution of citrate of ammonium, as proposed by Prof. P. Wagner. This method, together with an abstract of the results obtained by Wagner in the treatment of various phosphates, was presented by Dr. Shepherd in a late number of the American Chemical Journal, to which I will refer for the details of the method.

At my suggestion a comparison of this method with that at present approved by the Association of Official Agricultural Chemists was made by Mr. H. B. McDonnell in the laboratory of the Pennsylvania State College.

The results of his analyses of various phosphatic substances are presented in the appended table.

In determining Wagner's "soluble" acid (corresponding to our "available") duplicates of samples 1*a* to 6*a* were taken from different flasks, which stood about twenty hours over night, and were frequently shaken during the afternoon and morning. Samples 1*b* to 6*b* were treated similarly, but stood only six hours. The duplicates of 7*a* to 9*a* were taken from the same flasks and stood fourteen hours; 7*b* to 9*b* stood sixteen hours.

Owing to the pressure of other work, I am unable to do more than note the facts most clearly shown by the data.

Accepting the means of the results of 1*a* to 6*a* and 7*b* to 9*b* as those best representing Wagner's method, a comparison with the "available" acid obtained by the Association method shows that the former method gives somewhat higher results with fine ground South Carolina rock and fine ground bone, and more than doubles the amount in raw Navassa rock. On the other hand, it gives somewhat lower results with fine ground apatite, dissolved bone-black, and Orchilla guano, and several per cent. less with dissolved South Carolina rock, dissolved bone-ash, and dicalcium phosphate.

A comparison of the results in columns *a* and *b* to determine the limits of time in which the solution is completed, taking first samples 1*a* to 6*a*, shows that sixteen hours suffices to practically complete the solution of the available acid in fine ground apatite, fine ground bone, and dissolved South Carolina rock; and that there is a gain of 0.1 to 0.4 per cent. by standing twenty hours in the other cases. In samples 7 to

9 the results with Orchilla guano are not materially aided by an increase of two hours over the original fourteen hours allowed for solution, but in the other cases the gain is considerable.

Designation.	Association method.					Wagner's method.	
	Total P_2O_5.	Soluble.	Insoluble.	Reverted.	Available.	Soluble.	
						a.	*b.*
(1) Fine ground South Carolina rock	*Per cent.* 27.11	*Per cent.* .00	*Per cent.* 25.85	*Per cent.* 1.26	*Per cent.* 1.26	*Per cent.* 1.56	*Per cent.* 1.37
(2) Fine ground apatite	39.44	.00	38.59	0.85	0.85	0.66	0.63
(3) Fine ground bone.	24.06	0.16	19.33	4.57	4.73	5.19	5.28
(4) Dissolved South Carolina rock	17.19	10.04	3.50	3.65	13.69	11.56	11.49
(5) Dissolved bone-black	16.02	12.32	0.66	3.04	15.36	15.24	14.80
(6) Dicalcium phosphate	43.76	3.77	11.85	28.14	31.91	23.67	23.44
(7) Dissolved bone-ash..	21.87	14.29	3.61	3.72	18.01	14.39	14.74
(8) Raw Navassa.....	28.83	.00	27.80	1.03	1.03	2.42	2.51
(9) Orchilla guano.....	18.11	.00	14.43	3.69	3.69	3.25	3.27

The president then announced the subject as open to discussion.

Mr. CHAZAL remarked upon the uncertainty of determining specific gravity by means of the densimeter, except when great care is used in correcting the instrument, and hoped the determination by weight would be recommended.

Professors MOSES and LUPTON thought that the densimeter should be retained as a matter of convenience, but that careful correction should be made upon the instruments used.

Dr. JENKINS called attention to the fact that new thermometers of approved make are occasionally incorrectly graduated, and a thermometer correct when new will, on use, change its zero point very considerably, so that frequent examination is necessary to secure exactness as to temperature both in the preparation of ammonium citrate and in the extraction of citrate-soluble P_2O_5.

Prof. JOHN A. MYERS remarked that it is possible that much of the variation in the results of analysis may, no doubt, be accounted for by the fact that weights and other instruments used in the processes of analysis are subject to wide variations. Out of eight sets of weights in his laboratory bought for standard sets but three sets could be found that would compare sufficiently closely not to seriously effect the results of the work. Whether this trouble extends to other laboratories in the country he had no means of ascertaining.

The possible effects in the variation in the thermometers, and also in apparatus for determining specific gravity, may be seen in the difficulty of getting concordant results in our insoluble phosphate determinations, thus effecting the citrate of ammonium solution appreciably.

The opinion was finally elicited that members should use the methods which they preferred, but that great care should be used to insure accuracy.

Mr. CHAZAL then called attention to the loss occasioned when, in washing out soluble phosphoric acid, the fertilizer is washed from the funnel into a mortar and returned to the filter; also to the disadvantage of shredding filter paper into citrate solution causing danger of imperfect washing out with water and also retarding contact of fertilizer with the citrate solution when shaken.

He also brought up the question of the use of magnesic nitrate and showed the possibility of the retention of phosphoric acid in the hard white cake formed on ignition.

Dr. WHITE then read some notes of experiments on the use of Mg $(NO_3)_2$ in ignition.

ON THE USE OF MAGNESIUM NITRATE.

The following experiments were made to determine if ignition with Mg $(NO_3)_2$, in order to destroy organic matter, could be replaced by wet methods:

(1) A commercial fertilizer containing acid phosphate, potash salts, and animal matter was selected; total P_2O_5 was determined.

(*a*) By digesting 0.5 gram for thirty minutes with 30 cubic centimeters concentrated HCl.

(*b*) By digesting 0.5 gram for thirty minutes with 30 cubic centimeters concentrated HCl, adding at intervals pinches of finely powdered $KClO_3$ (0.5 gram in all).

(*c*) By digesting 0.5 gram for thirty minutes with 30 cubic centimeters fuming HNO_3.

(*d*) By igniting 0.5 gram with Mg $(NO_3)_2$ in usual manner.

Results were as follows:

	a.	*b.*	*c.*	*d.*
Total P_2O_5.....	11.68 11.73	11.95 11.98	11.82 11.80	12.05 11.93
Mean.....	11.70	11 97	11.81	11.99

(2) A commercial fertilizer containing acid phosphate, potash salts, and cotton seed meal was selected. Methods detailed above were employed. Results were as follows:

	a.	*b.*	*c.*	*d.*
Total P_2O_5.....	10.12 10.28	10.52 10.54	10.36 10.32	10.47 10.47
Mean.....	10.20	10.53	10.34	10.47

The results showed that the use of $KClO_3$ with HCl was much the best method of working, both in convenience and results.

The general testimony which followed was in favor of oxidation of the organic matter in other ways than by means of Mg $(NO_3)_2$.

Dr. JENKINS then brought up the subject of the Thomas-Gilchrist Schlage, which he said were beginning to be introduced into this country. As they contain phosphide of iron the phosphorus so combined would be oxidized by nitric acid and determined as phosphoric acid.

As the amount reaches 1 per cent. at times, this oxidation should be guarded against.

Dr. JENKINS also brought up the subject of the effect of the presence of fluorine on the determination of P_2O_5 in phosphates, such as Fluorapatite. Dr. WHITE said that the small amount of fluorine in Charleston rock had been found to make no difference.

Professor MYERS then asked in regard to the presence of silica in a soluble form.

Drs. JENKINS and WHITE and Professor STUBBS mentioned experiments in which the presence of silica to the extent of 10 per cent. had no effect. Dr. WHITE then contributed the following:

TO DETERMINE THE EFFECT SILICA IN SOLUTION HAS UPON MOLYBDATE DETERMINATIONS.

A highly silicious phosphate from South Georgia was used. It contained total silica, 10.76 per cent.

	Per cent.
Dissolved by boiling in strong HCl	0.11
Dissolved by ignition with Mg $(NO_3)_2$ and solution in strong HCl	0.21

(*a*) 0.5 gram was digested with 30 cubic centimeters of concentrated HCl, diluted, allowed to settle, poured off, the residue repeatedly boiled with strong HCl, the fluids mixed, and P_2O_5 determined.

(*b*) 0.5 gram was ignited with Mg. $(NO_3)_2$ dissolved in strong HCl, as above, and P_2O_5 determined.

(*c*) 0.5 gram was treated as in (*b*), an aliquot of the filtrate was evaporated to remove silica in usual manner, and P_2O_5 determined. Results were as follows:

	a.	*b.*	*c.*
Total P_2O_5	22.54 22.58	22.65 22.60	22.59 22.61
Mean	22.56	22.63	22.60

The indications are that the small percentages of silica brought into solution in the ordinary work of members of this association do not appreciably affect the results in P_2O_5. It may be assumed that it is not necessary to rid the solutions of silica.

Dr. GASCOYNE found that the presence of silica had no effect on the determination of phosphoric acid by the molybdate method.

An experiment with a mixture of nine parts of acid phosphate and one part finely ground silica gave 14.93 per cent. of P_2O_5 as the average of five determinations by the official method, and 14.92 per cent. P_2O_5 as the average of five determinations of the nitric acid method. Three determinations, made by evaporating to dryness, igniting, and redissolving in HCl, gave 2.89 per cent. as the average.

In discussion of these papers on the use of nitric acid instead of hydrochloric acid and magnesium nitrate, Dr. JENKINS said:

The reasons which led to the adoption of hydrochloric acid as a solvent and magnesium nitrate as an oxidizing agent are the following: Hydrochloric acid dissolves all phosphates more rapidly, though not

more completely than nitric acid, if sufficient pains are taken with the latter, but less time and watchfulness are needed when HCl is used, and for these reasons it has the preference. I admit that in many cases, particularly where alkaline salts are present, as in the ordinary grades of superphosphates simple ignition will remove organic matter and leave all the phosphoric acid in the tribasic or ortho-form. But in some cases it will not—as, for instance, in the case of South Carolina superphosphate in Dr. White's interesting table. Now our uniform method must cover all cases likely to occur, and therefore some basic oxidizing mixture must be used unless organic matter can be destroyed in the wet way. Magnesium nitrate is the best mixture of the kind, for it does not flux silica. Dr. White's experiments, however, prove, I think, that the organic matter can be destroyed or sufficiently altered for our purpose in the wet way, and as that method is much simpler I favor its adoption.

Professor STUBBS remarked that by accident it was found in his laboratory that HNO_3 could supplant $Mg(NO_3)_2$ in analyses of complete fertilizers. The fertilizers, consisting of cotton-seed meal, acid phosphate, and kainite, were ignited with HNO_3 and analyzed. Being afraid of error, they were duplicated with $Mg(NO_3)_2$ and the results found to agree.

After some further remarks the committee was directed to prepare the outline of a method to be used during the ensuing year.

By unanimous consent it was then resolved that the president should appoint a committee to consider the advisability of extending the scope of the Association, to report on Friday.

The president appointed Dr. White, chairman, Dr. Jenkins, and Dr. McMurtrie. The convention then adjourned till afternoon.

THURSDAY AFTERNOON.

The convention having been called to order,

Mr. CHAZAL moved, and, after discussion, it was adopted, that the samples distributed by the committees be sent out on or before October 1, and that results be reported before July 1, the work upon phosphoric acid to be done, when possible, at some definite time designated by the committee.

The report of the Committee on Potash then being in order was then submitted by the chairman, Dr. Gascoyne:

REPORT OF THE POTASH COMMITTEE.

As required by the constitution of the Association the Committee on Potash herewith presents a brief report on work done during the past year. Last January your

committee sent six samples containing potash to fifteen official and commercial chemists, accompanied by the following circular letter:

"RICHMOND, VA., *January* 6, 1886.

"DEAR SIR: In behalf of the committee on potash appointed by the Association of Official Agricultural Chemists at the Washington meeting last September, I send you by express six samples containing potash, numbered 1 to 6, respectively.

"Please determine in them potash by the method adopted by the association at the Washington meeting and also by any other method you may desire to try. When the work is done please report results to me with detailed accounts of methods employed other than that adopted by the Association.

"Very truly, yours,

"W. J. GASCOYNE,
"*Chairman Potash Committee.*"

In the preparation of these samples chemically pure potassium salts were used, the materials very carefully mixed, and the samples sent to the members in sealed bottles.

The following is a description of the samples:

No. 1. Potassium sulphate, C. P.

No. 2. Commercial kainite.

No. 3. Acid phosphate, with pure potassium sulphate, sodium chloride, and magnesium sulphate, containing 2.70 per cent. of potash.

No. 4. Acid phosphate, with pure potassium sulphate, potassium chloride, and potassium nitrate, containing 4.09 per cent. of potash.

No. 5. Acid phosphate, with potassium sulphate, sodium chloride, and cotton-seed meal, containing 3.19 per cent. of potash.

No. 6. Acid phosphate, with potassium sulphate, potassium chloride, and sodium nitrate, containing 2.25 per cent. of potash.

The results are given in two tables, the first of which gives the results by the association method, and the other by the method suggested by Mr. T. S. Gladding at the last meeting of the association.

Potash determinations.

ASSOCIATION METHOD.

Analyst.	K_2O No. 1.	K_2O No. 2.	K_2O No. 3.	K_2O No. 4.	K_2O No. 5.	K_2O No. 6.
A	53.99	12.86	2.75	3.95	3.26	2.19
B	54.09	13.11	2.56	3.80	2.61	1.20
C	54.02	13.05	2.68	3.92	3.20	2.21
D	53.76	13.33	2.71	3.79	3.18	2.17
E	53.88	13.42	2.71	3.75	3.07	2.19
F	53.98	13.04	2.57	3.91	3.20	2.20
G	54.00	13.32	2.76	3.87	3.50	2.34
H	54.08	13.65	2.58	3.89	3.22	2.13
I	53.12	13.43	3.10	4.05	3.59	2.62
J	53.88	....	....	....	....	....
K	53.33	15.46	2.57	3.76	2.71	2.05
L	53.96	12.06	2.42	3.40	1.95	1.86
Average	53.88	13.33	2.67	3.83	3.03	2.10

LINDO-GLADDING METHOD.

C[1]	53.96	12.95	2.74	3.86	3.13	2.19
L[1]	53.49	12.67	2.41	3.63	2.89	1.94
M	54.00	13.05	2.54	3.79	3.13	2.26
Average	53.82	12.89	2.56	3.76	3.05	2.13

In the table below are given the average results by the Association method, with the highest and lowest determinations:

	Average.	Highest.	Lowest.	Difference.
No. 1	53.88	54.09	53.12	.97
No. 2	13.33	15.46	12.06	3.40
No. 3	2.67	3.10	2.42	.68
No. 4	3.83	4.05	3.40	.65
No. 5	3.03	3.59	1.05	1.54
No. 6	2.10	2.62	1.20	1.42

In results by the Lindo-Gladding method, in sample No. 1, the difference between the highest and lowest determination is .51 per cent.

In No. 2 the difference between the highest and lowest determination is .38 per cent.

In No. 3 the difference is .33 per cent.

In No. 4 the difference is .23 per cent.

In No. 5 the difference is .24 per cent.

In No. 6 the difference is .32 per cent.

The report of the committee having been accepted, it was moved that the place of N. W. Lord, who was not present, be filled by the president.

He named Prof. M. A. SCOVELL, of Kentucky.

Discussion being in order, Professors MCMURTRIE and CHAZAL stated their objections to the use of a porcelain dish for driving off ammonia salts and organic matter.

Professor SCOVELL said: Mr. President, I wish to call attention in the official method of estimating potash to the use of a porcelain dish in which the potash solution is brought to dryness and the ammonic salts volatilize. In my own case I find it almost impossible to volatilize all the ammonia salts in a porcelain dish below a red heat, especially if not redissolved and reignited, this portion of the method is apt to mislead, and I would suggest the use of a platinum dish instead of porcelain. Unless great care is taken the results will be too high by the use of a porcelain dish.

Professors WHITE and MOSES called attention to the difficulty of washing the barium precipitate and the necessity of using boiling water repeatedly.

Dr. JENKINS said: A source of error in potash determination is this. In presence of a considerable quantity of alkali chlorides it is impossible perfectly to separate magnesia by barium hydrate, and if insufficient platinum chloride is used, the results obtained may be too high. The error may be avoided by adding in every case a very considerable excess of platinum solution.

Another and more common source of error is imperfect washing of the precipitate produced by $BaCl_2$ and $Ba(OH)_2$. It is well known that $BaSO_4$ is apt to carry down with it soluble salts and the gelatinous precipitate of barium phosphate is also hard to wash. I always use from 350 to 500 cubic centimeters of wash water, washing several

times by decantation with boiling water before bringing the precipitate on a filter.

Professor CHAZAL objected to the fact that the use of the Gooch crucible should be required, and desired that the method of filtration should be optional.

Professor MOSES considered washing the double salt through the filter with hot water and afterwards evaporation and weighing as being as convenient as the Gooch crucible.

Dr. GASCOYNE called the attention of the members to the atomic weight of platinum by W. Halberstadt in Zeit. für anal. Chem., Heft 2, 1886.

Halberstadt gives as the mean of about 100 determinations 194.5759 as the atomic weight of Pt, thereby confirming the atomic weight as given by Seubert.

He also spoke upon the Lindo method as modified by Gladding, and recommended its examination by chemists, being confirmed by Mr. Richardson, who thought the use of the half-saturated solution of NH_4Cl according to Gladding's method would be found advantageous in any case. Dr. White suggested that the Gladding method be reported as an alternative, and also brought up the question of precipitating with barium chloride, hydrate and ammonic carbonate in an original portion of two grams of the fertilizer without intermediate filtration.

Dr. WILEY said that on page 32 of the Proceedings for 1885 this had been abandoned.

Mr. RICHARDSON asked for opinions in regard to use of oxalic acid.

Dr. JENKINS saw no necessity, and gets higher results in kainite without it.

Professor MYERS said that he had made some comparative analyses, possibly thirty or forty determinations with the use of oxalic acid and without, using simply carbonate of ammonium.

Without having the figures with him he would state that the results appear to be equally accurate, except in the case of kainite, in which there is trouble unless oxalic acid is used, and even this fails, without care, to remove the impurities.

Dr. GASCOYNE found it useful in removing something not precipitated by ammonic carbonate.

Professor SCOVELL thought the evaporation after addition of platinum should be carried further than a sirup, to remove all HCl, as he considered the double salt somewhat soluble in this.

Dr. JENKINS said he found there was no appreciable effect from this cause, but that going to dryness would produce no injury. The concentration of the alcohol used for washing was not vital.

On a question from Mr. Richardson, no objection was raised to the use of the old factors for the conversion of K_2PtCl_6 to K_2O.

The convention then adjourned until Friday.

FRIDAY MORNING.

The convention was called to order at 10 o'clock.

The order of business being the continuation of reports of committees.

The secretary, in the absence of Dr. Dabney, read the following:

RALEIGH, N. C., *August* 24, 1886.

GENTLEMEN: Your Committee on Nitrogen esteemed it most important to settle upon a general method for determining nitrogen in the course of fertilizer work. To this end they thought that the best plan was to send out a well-defined method along with the samples, which was to be used by all of the analysts, and compared with such other methods as they choose to try.

It was desirable that the method thus put on trial should be applicable to all the ordinary sources of nitrogen, simple, easy, and rapid of execution in the most ordinary laboratory, and that it should promise to give results agreeing fairly with those obtained by the so-called absolute method, when carried out with the best apparatus and manipulation.

The committee selected the Ruffle method as promising the most by this standard, and sent out a description of it to your members.

The results on the first samples were so late coming in that we were unable to send out other samples and another method, as we expected to do.

We have the honor of submitting herewith the results obtained by the Ruffle method, as described, and by some other methods, upon four samples of different kinds.

These results are encouraging. Omitting two or three very out-of-the-way results, which are clearly due to accidents, the figures agree very well. The committee's Ruffle method gives results on the different materials which agree remarkably well.

Your committee suggests, therefore, the adoption of the Ruffle method, on trial, for general fertilizer work for one year. You will in this case, we hope, prepare detailed directions for carrying out the said method.

Respectfully submitted.

CHARLES W. DABNEY, Jr., *Chairman*,
W. E. MOSES,
JOHN A. MYERS,
Committee.

To the OFFICIAL AGRICULTURAL CHEMISTS' ASSOCIATION.

RESULTS OF THE DETERMINATIONS OF NITROGEN IN THE SAMPLES SENT OUT BY THE NITROGEN COMMITTEE.

(1) An ordinary ammoniated superphosphate of the trade.

(2) An ordinary ammoniated superphosphate of the trade.

(3) Cotton-seed meal.

(4) Three parts sugar c. p. to 1 part nitre c. p. calculated 3.47 per cent. nitrogen, or 3.38 nitrogen by the absolute method.

Results expressed as nitrogen.

BY THE COMMITTEE'S RUFFLE METHOD.

Analyst.	A.	B.	C.	D.	E.	F.	G.	H.	I.
1	1.78	1.85	1.66	2.10	.....	1.66	1.93	1.80	1.69
2	2.04	2.15	2.00	1.99	.....	2.07	1.80	2.12	1.92
3	7.45	7.53	7.43	7.56	.....	7.48	7.05	6.35	7.50
4	3.36	3.40	3.30	2.45	3.37	3.49	3.45	3.44	3.05

Results expressed as nitrogen—Continued.

BY THE KJELDAHL METHOD.

Analyst.	A.	B.	C.	D.	E.	F.	G.	H.	I.
1		1.85		2.04		1.79			
2		2.17		2.01		1.99			
3		7.33		7.44		7.40			
4						*2.38			

*2.76 with benzoic acid: 2.41 with sucrose.

BY THE SODA-LIME METHOD.

1	1.84	1.68		1.84		1.43			
2	2.05	1.96		1.95		1.54			
3	7.25	7.37	7.58	7.37		6.38			
4	2.10					2.40			

THE RUFFLE METHOD FOR NITROGEN, AS PROPOSED BY THE COMMITTEE.

Reagents:

(1) Standard Sol. H_2SO_4. .019968 gr. SO_3 to 1 cubic centimeter. Everything depends on the correctness of this solution.

(2) Standard Sol. KOH of exactly same strength as the acid.

(3) Soda lime crushed so that one-half is powder and one-half in granules.

(4) Hyposulphite soda coml. fused, almost water free and powdered.

(5) Charcoal and sulphur, equal parts by weight of flowers of sulphur and finely powdered wood charcoal (N-free).

(6) Saturated Sol. of "coralline" in alcohol.

Apparatus:

(1) Combustion furnace.

(2) U tube with stopcock.

(3) Two gallon bottles with syphon to aspirate.

(4) Combustion tube of best Bohemian glass, drawn to point, length 17½ in.; outside diameter ⅝ inch, inside diameter ½ inch.

Preparation:

(1) Clean and fill U tube with 10 cubic centimeters of SO_3. solution (or in proportion to the N. expected).

(2) Fit cork and glass connecting tube, (3), taking two sheets of letter-size paper No. 1 and No. 2; place on No. 1 two grams powdered hypo., on No. 2 equal portions of soda lime and hypo., and mix. This mixture should be enough to fill 9 inches of the tube or about 15 to 20 grams of each ingredient. Fill the tube as follows:

(1) A plug of asbestos.

(2) One to one and a half inches of hypo. and soda lime mixture from No. 2 paper.

(3) Mix intimately 1.4 grams (or .7 or 2.8 grams, as is thought best) of the substance to be analyzed with the powdered hypo. on No. 1; then add to the same 2 grams of the C. and S. mixture; mix these thoroughly; next pour on No. 1 the mixture from No. 2, except enough to fill 1½ inches of the tube; then mix carefully the mass now on No. 1 and pour into the tube; rinse off paper No 1 with balance of hypo. and soda lime from No. 2.

(4) Knock down the contents of the tube as far as they will go; then further rinse off No. 1 with pure soda lime, and pour into the tube to within 1½ inch of the end. (Space for swelling of the mass.)

(5) Another plug of asbestos and cork.

(6) Holding the filled tube nearly horizontal, tap on table until there is a gas channel all along the top of tube as it will lie in furnace. Place in furnace and make connections. Aspirate and see that all is tight.

The combustion:

Commence by heating the pure soda lime portion until it is a good red heat; then turn up slowly jet after jet toward the other end of the tube, so that the bubbles come off two or three a second. When the whole tube is red hot and all bubles cease and the liquid in the U tube begins to recede towards the furnace lower the flames somewhat, break off end of tube, and aspirate to extent of about one-half liter. Detach U tube, wash contents into a beaker, add five drops coralline solution and titrate.

Follow this scheme closely on committee's samples, criticise it, and suggest changes.

The secretary then read a paper by Mr. G. L. SPENCER, on

THE KJELDAHL METHOD FOR THE DETERMINATION OF NITROGEN.

This method for nitrogen determinations has attracted considerable attention since it was announced some three years ago. It is of special interest to agricultural chemists, affording as it does a rapid method for the determination of nitrogen in most of its combinations. It has even been claimed that the nitrogen of nitrates can be determined in this way. The results of a large number of analyses of pure nitrates made in this laboratory indicate that this claim is an extravagant one. This will be discussed further on.

The original method as described by Kjeldahl* has been considerably improved and its application extended by other chemists, notably by Wilfarth,† and later by Asboth.‡ As these modifications are published in detail in the journals referred to, it is not necessary to repeat them here. The following is an outline of the method which I adopted. It is essentially that of Asboth.

Take from .25 to 1 gram of the substance under examination. Introduce it into a flask (500 cubic centimeters); add .5 to 1 gram anhydrous sulphate of copper, 1 gram of mercury, and finally 15 cubic centimeters of the following mixture:

100 grams sulphuric acid.
50 grams phosphoric anhydride.

Heat at a temperature a little below the boiling point of the mixture. Half an hour is usually long enough to continue the heat. After thorough decomposition the contents of the flask are cooled, 50§ cubic centimeters water are added and the solution is again cooled. One hundred and twenty cubic centimeters of the following solution are added:

Rochelle salt	grams	350
Sodium hydrate	do	300
Water	liter	1

As soon as sufficient sulphide of potassium to precipitate the mercury and a small piece of zinc have been added the mixture is ready for distillation. Distil over about 50 cubic centimeters into a standard acid solution. Titrate as usual.

I find that the addition of zinc is necessary to prevent bumping. By passing the vapors through a bulb containing broken glass all liability to error from portions of the alkali being carried over with the liberated hydrogen is obviated. I prefer to have the bulb at the end of the tube projecting into the distillation flask. This is essentially the plan recommended by Pfeiffer‖ and Lehmann. A number of blank experiments demonstrated that there is no error from this source when this device is employed.

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* Zeit. anal. Chem., 1883, p. 366.

† Wilfarth's method, Chem. Centralblatt, 1885, p. 17.

‡ Asboth's method, Chem. Centralblatt, 1886, p. 161.

§ 100 cubic centimeters in the original method.

‖ Bulletin No. 7, Division of Chemistry, U. S. Dept. of Agrl., p. 43.

The following table gives a record of the experiments and the results obtained:

Nitrogen determinations by Kjeldahl's method.

Substance.	Number.	Serial number.	Per cent. of nitrogen.	Trescot's results.				
				Average.	Soda lime.		Ruffle.	
					Per cent. of nitrogen.	Average.	Per cent. of nitrogen.	Average.
Milk	*a*1	.....	.543	.543	.459	.459	.....	.....
Do	*a*2	.....	.459	.459	.465	.465	.....	.....
Do	*a*3	.....	.448	.448	.426	.426	.....	.....
Do	*b*4	.....	.448	.448	.502	.502	.....	.....
Do	*b*5	.....	.498	.498	.409	.409	.....	.....
Mustard	*b*6	4511	6.08	.....	5.98	.....	.....	.....
Do	*c*7	4511	6.38	.....	5.98	.....	.....	.....
Do	*b*8	4511	6.27	.....	5.98	.....	.....	.....
Do	*b*9	4511	6.19	.....	5.98	.....	.....	.....
Do	*b*10	4511	6.33	6.25	5.98	5.98	.....	.....
Do	*d*11	4542	4.20	.....	4.05	.....	.....	.....
Do	*d*12	4542	4.48	.....	4.05	.....	.....	.....
Do	*d*13	4542	4.26	....	4.05	.....	.....	.....
Do	*d*14	4542	4.26	4.30	4.05	4.05	.....	.....
Do	*e*15	4542	4.83	.....	4.05	.....	.....	.....
Do	*e*16	4542	4.84	.....	4.05	.....	.....	.....
Do	*e*17	4542	4.79	4.82	4.05	4.05	.....	.....
Fertilizer	*d*18	4447	7.73	.....	6.77	.....	.....	.....
Do	*d*19	4447	7.70	.....	6.77	.....	.....	.....
Do	*d*20	4447	7.64	.....	6.77	.....	.....	.....
Do	*d*21	4447	7.78	7.71	6.77	6.77	.....	.....
Pepper	*e*22	4537	2.10	.....	1.79	.....	.....	.....
Do	*f*23	4537	2.07	2.09	1.79	1.79	.....	.....
Cotton-seed meal	*f*24	4194	7.39	.....	6.35	.....	.....	.....
Do	*f*25	4194	7.36	7.37	6.35	6.35	.....	.....
Casein	*f*26	.....	13.44	.....	13.07	.....	.....	.....
Do	*f*27	.....	13.55	13.49	13.11	13.09	.....	.....
Gluten	*f*28	.....	16.02	.....	16.23	.....	.....	.....
Do	*f*29	.....	15.90	15.96	16.16	16.19	.....	.....
Fertilizer	*f*30*	4450	1.79	.....	1.46	.....	1.68	.....
Do	*f*31	4450	1.79	1.79	1.40	1.43	1.62	1.65
Do	*f*32*	4451	2.02	.....	1.51	.....	2.07	.....
Do	*f*33	4451	1.96	1.99	1.57	1.54	2.07	2.07
Cotton-seed meal	*f*34*	4452	7.42	.....	6.38	.....	7.48	.....
Do	*f*35	4452	7.39	7.40	6.38	6.38	7.48	7.48
Fertilizer	*f*36*	4453	2.35	.....	2.02	.....	3.42	.....
Do	*f*37	4453	2.41	2.37	2.05	.....	3.58	.....
Do	*g*38	4453	2.80	.....	2.13	.....	3.58	.....
Do	*g*39	4453	2.72	2.76	2.24	.....	.....	.....
Do	*h*40	4453	2.41	2.41	2.40	2.17	.....	3.53

a One-half gram Cu. SO_4; the water foamed badly.
b One-half gram Cu. SO_4; 100 cubic centimeters water+Zn. foamed badly.
c As above, except in Cu. SO_4+Zn.
d One-half gram Cu. SO_4; 50 cubic centimeters H_2O.
e One-half gram Cu. SO_4; 50 cubic centimeters H_2O; 1 gr. Hg, K_2S to precipitate Hg.
f Same as *e* except+Zn.
g Same as *f*, except+benzoic acid.
h Same as *f*, except+pure sucrose.
* Samples furnished by the committee.

An inspection of the table shows that the results obtained were more uniform when mercury was employed.

The following results were obtained with C. P. ammonium salts:

Substance.	Kjeldahl.	Will and Warrentrapp.	Theoretical.
Ammonium chloride	26.432	26.210	26.168
	26.430	26.210	26.168
	26.580	26.210	26.168
Ammonium oxalate	19.570	19.710	19.718
	20.220	19.710	19.718
	20.220	19.710	19.718
	20.040	.....	19.718

I cannot account for the high percentages obtained with these salts. The combustion with soda-lime gives results varying but little from theory.

In his paper,* Dr. Asboth recommends the addition of benzoic acid to the decomposing mixture when nitrates are present. Kjeldahl does not claim that the method is applicable to nitrates.

I have made a number of experiments by the method as suggested by Dr. Asboth. The results given in the following table show that with this modification the work was very unsatisfactory:

Substance.	Kjeldahl.	Ruffle.	Theoretical.
Pure nitrate of sodium, Com'l.	9.24	.....	16.47
	9.21	15.65	16.47
	13.33	16.16	16.47
	13.33	.....	16.47
	10.47	.....	16.47
	11.20	.....	16.47
	10.53	.....	13.47
Pure nitrate of potassium....	13.00	13.55	13.84
	12.94	13.66	13.84
	11.89	14.00	13.84

The unsatisfactory results obtained by the Kjeldahl and Ruffle methods with nitrates led me to experiment upon a method to accurately and quickly determine the nitrogen of nitrates and other forms in the same substance.

Not having had time to make but very few experiments I shall only outline the method and results.

Make an ordinary nitric acid determination by Schulze's method as modified by Tiemans. (See Fresenius Quant. Anal., p. 473.) Instead of employing ferrous chloride, as directed, I use the sulphate, which answers equally as well and is more convenient.

Transfer the residue from the nitric acid analysis to schälchen and evaporate to dryness, or dry in the flask used for determining the nitric acid. The nitrogen in the residue is determined by the Kjeldahl method. The nitric acid analysis can easily be made in three-quarters of an hour.

A number of experiments made on mixtures of nitrates and cotton-seed meal, gluten, &c., gave nearly theoretical results. It is possible that with very volatile ammonium compounds there might be a loss of ammonia in the nitric-acid determination.

At some future time I hope to continue these experiments and determine the value of this combination of the two methods.

CONCLUSION.

The results obtained (except with nitrates) and the facility with which analysis can be made, recommended the Kjeldahl method to chemists for a large proportion of nitrogen work usually done in agricultural laboratories.

References.—Chemisches Centralblatt, 1885, p. 17; Chemisches Centralblatt, 1886, p. 161; Zeit. annal. Chem., 1883, p. 366; Bulletin Soc. Chemie, vol. 44, p. 623; Chemical News, vol. 52, p. 162; Chemical News, vol. 52, p. 220; Chemikerzeitung, 1885, p. 780; Chem. Centralblatt, 1885, p. 585.

Dr. GASCOYNE presented the following table, giving the results obtained on a variety of fertilizing material by the soda-lime method; the method suggested by Professor Johnson, using slaked lime as a substitute for soda lime; the Ruffle method, and a modification of the Ruffle method, using slaked lime instead of soda lime, described in full in

* Chemical Centralblatt, 1886, p. 161.

the report of the nitrogen committee on methods to be used for the determination of nitrogen during the next year.

	Soda lime.		Slaked lime.		Ruffle method.		Ruffle method modified.	
	Per ct.	*Average.*	*Per ct.*	*Average.*	*Per ct.*	*Average.*	*Per ct.*	*Average.*
Ammoniated superphosphate	1.67 1.69	1.68	1.64 1.68	1.66	1.63 1.69	1.66	1.66 1.70	1.68
Do	1.94 1.98	1.96	1.97 1.99	1.98	1.97 2.03	2.00	1.97 2.01	1.99
Bone meal	4.10 4.14	4.12	4.07 4.11	4.09	4.13 4.17	4.15	4.15 4.15	4.15
Cotton-seed meal	7.35 7.39	7.37	7.30 7.36	7.33	7.40 7.46	7.43	7.38 7.42	7.40
Dried blood	12.23 12.31	12.27	12.27 12.31	12.29	12.31 12.39	12.35	12.33 12.41	12 37
Azotin	12.98 13.04	13.01	12.98 13.02	13.00	13.03 13.09	13.06	13.05 13.11	13.08
Fish scrap	8.12 8.18	8.15	8.13 8.17	8.15	8.14 8.22	8.18	8.12 8.18	8.15
Castor pomace	4.98 5.04	5.02	4.98 5.02	5.00	5.00 5.04	5.02	5.00 5.06	5.03
Peruvian guano	7.46 7.50	7.48	7.45 7.49	7.47	7.51 7.55	7.53	7.50 7.56	7.53
Ammoniated superphosphate with 1 per cent. of nitrogen from nitrate	.....	.....	.....	.....	4.30 4.36	4.33	4.32 4.40	4.36
Ammoniated superphosphate with 1 per cent. of nitrogen from nitrate	.....	.....	.....	.....	6.44 6.52	6.48	6.44 6.48	6.46
Ground tobacco stems	.....	.....	.....	.....	1.54 1.58	1.56	1.50 1.54	1.52
Commercial nitrate of soda	.....	.....	.....	.....	15.90 15.96	15.93	15 91 15.99	15.95

Dr. GASCOYNE stated that he had made a number of tests by the modified Ruffle method, using potasium xanthogenate as a substitute for the charcoal and sulpur mixture with good results.

Discussion of the report and papers being in order,

Dr. JENKINS gave it as his experience that the Kjeldahl method gave identical results with the absolute method in the absence of nitrates. Coarse bone, high grade blood, and wheat bran gave results from one to three-tenths per cent. higher than with soda lime. He had also obtained good results with ferro- and ferri-cyanide of potassium and found that on pure ammonia salts the absolute and Kjeldahl methods agreed. A source of error to be avoided in the latter method was the presence of ammonic sulphate in the sulphuric acid used.

Dr. CHAZAL considered the committee's samples as an unfair test of the Ruffle method, as they contained nitrate in only one instance.

Professor LUPTON said: We have found in my laboratory the Kjeldahl method unreliable in the case of nitrates, but satisfactory in other cases. One great objection to the method is the length of time required for digestion with sulphuric acid. From twelve to fifteen hours were necessary in the case of the samples sent out by the committee.

Dr. GASCOYNE then spoke in favor of the use of dry slaked lime and crystallized hyposulphite instead of the committee's mixture; and

Dr. WHITE expressed his approval of the former, using enough sodic hydrate, however, to make the lime more compact and granular.

It was then resolved that in the absence of Dr. Dabney, the Chair should appoint some one to fill his place to act with the remainder of the committee in preparing recommendations for the ensuing year.

Dr. Gascoyne was appointed.

While awaiting the recommendations of the several committees as to methods to be adopted by the Association, by unanimous consent Mr. de Ghequier was allowed to address the convention.

Mr. de Ghequier read the following letter:

THE NATIONAL FERTILIZER ASSOCIATION,
Baltimore, August 4, 1886.

THE OFFICIAL CHEMISTS' ASSOCIATION,
Washington, D. C.:

The undersigned, forming the committee on legislation of the National Fertilizer Association, herewith request you, by passage of a resolution of your association to that effect, to approve the pending amendment of Senator Chace, of Rhode Island, to Senate bill No. 372, which is likely to come up early in the next session of Congress.

Yours, very respectfully,

JOHN M. GLIDDEN,
President and Chairman, Pacific Guano Company, Boston, Mass.
SAMUEL IVERS,
Clark's Cove Guano Company, New Bedford, Mass.
J. B. E. SLOAN,
Edisto Phosphate Company, Charleston, S. C.
J. C. GRAFFLIN,
Navassa Phosphate Company, N. Y., Baltimore, Md.
E. FRANK COE,
16 *Burling Slip, New York City.*

A. DE GHEQUIER, *Secretary.*

And then said:

GENTLEMEN: I have the honor to respectfully submit on behalf of the National Fertilizer Association this request of the committee on legislation, to the effect that you may be pleased to pass before adjourning, a resolution by which as an association of official chemists, you will indorse the incorporation of such amendment, to Senate bill 372 (H. R. 2933) as will secure the adoption of that method of analysis which you from time to time may decide upon, in all States desiring to obtain the appropriation of $5,000 per annum for either agricultural experiment stations or agricultural colleges, and secure likewise one uniform mode of regulation brand, and, above all, uniformity in branding bags, together with an abolition of commercial valuations. Our Association from its close intercourse with the members of your Association thoroughly appreciates the sentiments which from time to time have been expressed by you on this very subject, and because it is aware of the fact that your views coincide fully on this matter with those entertained by the trade, the committee on legislation indulges in the hope that the request now preferred to put your opinions in a tangible form on record so as to enable us to urge on solid grounds the passage of such amendments as will procure for the trade the possibility to have for one brand only one bag

will meet with a favorable reception. The multiplication of State laws increases yearly the number of bags a manufacturer has to keep on hand, and exposes him uselessly to lawsuits and losses without any benefit whatsoever accruing from this variety of expressions to the farmer. On the contrary, the views of the vast majority of the members of your Association clearly indicate that you not only recognize the justice of our desire to see an absurdity replaced by a provision more in accord with the dictates of common sense, but are disposed to assist us in this direction. The opportunity to do so in an efficient way now presents itself, and hence the request of our Association for the passage of a resolution indorsing Senator Chace's amendment.

Nearly 10,000,000 bags are consumed by the trade, and some firms use upwards of 300,000. For four brands four bags would suffice; at present, however, it is necessary to keep a stock at least sixty to a hundred on hand. It is a hardship the greatness of which no one can properly realize except those who during the rush of a shipping season witness the trouble and annoyance which is daily incurred on that ground.

The very science—agricultural chemistry—which has called the fertilizer industry into being, should certainly not be the means of throwing superfluous obstacles in the way of the prosecution of that business, and the time has arrived, it seems to me, when to the signal services rendered by agricultural chemistry to the trade in a scientific way proper assistance in a business way should be granted by removing what you yourselves admit to be entirely useless obstructions. As regards commercial valuations, it will ever remain an impossible attempt to impress upon the farmer that they represent only a means of comparing prices, but do not represent either the agricultural value of fertilizers or the figure at which a manufacturer should sell his goods. It is carrying the paternal government theory too far, and experience has shown that no good has thereby been accomplished. If the farmer is incompetent to judge from the analysis what is and what is not a good article, why is his ignorance not in a similar way assisted in the many other articles which he is bound to purchase. Flour, agricultural implements, boots and shoes, prints, &c., might, with precisely the same justice, and on precisely the same ground, have commercial valuations attached, and I think it would be quite a surprise to farmer and scientist to see what an enormous margin of profit there is in nearly every commodity and how comparatively insignificant the same margin is on fertilizers. Commercial valuations can hardly be said to be more than a discrimination against the very industry which has done more to build up a greater productiveness of the soil than all others put together. On this point, however, there has so much been said that is quite familiar to you that I forego to dwell upon this subject.

Our Association is in hearty accord with the bill appropriating $5,000 per annum to each State, because it believes that these additional funds will enable the Control Stations to increase their usefulness and lighten,

by the employment of an increased force, the labor which at certain seasons renders the chemist's work in the laboratory a very heavy and trying burden.

The publication of the method of analysis in all the minutiæ of detail goes far towards reducing differences, as it enables the chemists employed by the manufacturers to conform to it as that the official analyses ought to be only confirmations of those previously obtained by the manufacturer. I am sure the trade will think the millennium has arrived if the discrepancies are once reduced to three-tenths of 1 per cent., and from the results laid before this meeting by the several committees it would seem as if that day is near at hand.

Permit me in conclusion to thank you for the interest you take in, and the unpaid labor you bestow upon, those investigations which in the end tend to benefit not only science but the fertilizer industry likewise, and to tender you also, on behalf of the committee on legislation and of the Association, their grateful appreciation of the consideration you accord to our representatives and requests.

In reply Professors STUBBS and LUPTON spoke strongly against the Association taking part in any national legislation. They were willing to do everything possible with their State legislatures, but could not feel justified in sanctioning national interference in the matter.

After some further remarks from other members of the Association, on motion of Dr. Jenkins the communication of Mr. de Ghequier was referred to the Committee on Uniformity in Legislation.

The Committee on Revision of the Constitution, with a view to enlarging the field of the Association, was then asked to report, and submitted the following, which was unanimously adopted:

The committee appointed to revise the constitution, in accordance with the suggestions referred to it, recommends the following changes:

Section 1 to read:

(1) This Association shall be known as the Association of Official Agricultural Chemists of the United States. The objects shall be (1) to secure uniformity and accuracy in the methods, results, and modes of statements of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

Section 2 to read:

(2) Analytical chemists connected with departments of agriculture, agricultural experiment stations, agricultural colleges, State boards of agriculture, and other bodies charged with official control of the materials named in section 1, shall alone be eligible to membership, and one such representative for each of these institutions or boards when properly accredited shall be entitled to a vote in the Association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. Any person eligible to membership may become a member at any meeting of the Association by presenting proper credentials and signing this constitution. All analytical chemists and others interested in the objects of the Association may attend its meetings and take part in its discussions, but shall have no vote in the Association.

Section 4 to read:

Strike out the word "three" in first line. Insert after "Committee on Nitrogen"

the following: "The committee on cattle foods and the committee on dairy products, whose reports shall be taken up, discussed, and disposed of in the order named. Additional committees may be appointed from time to time as the Association may direct."

Section 7 to read:

Seven enrolled members, entitled to vote, shall constitute a quorum for the transaction of business, instead of a "majority shall constitute a quorum."

Section 9 to be added:

All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by a vote of two-thirds of the members present and entitled to vote.

The remaining portions of the constitution, as now printed, to be retained unchanged.

Respectfully submitted.

H. C. WHITE, *Chairman.*
E. H. JENKINS.
WM. McMURTRIE.

The Association then proceeded to the election of a committee to nominate officers for the ensuing year, and Messrs. LUPTON, STUBBS, and SCOVELL were chosen.

After withdrawing for a short time they reported as follows:

The committee appointed to nominate officers for the next year respectfully report that they are unable to agree, and recommend that officers be elected by ballot without nominations and the committee be discharged.

N. T. LUPTON, *Chairman.*
W. C. STUBBS,
M. A. SCOVELL.

The recommendations were accepted but not adopted, and the committee discharged, and it was voted that the Chair appoint another committee.

Professors GASCOYNE, MOSES, and COOKE were appointed.

The convention then adjourned until 2.30 p m.

FRIDAY AFTERNOON.

The convention having been called to order, Dr. WHITE reported as follows:

The Committee on Phosphoric Acid has carefully considered the several suggestions referred to it, and respectfully recommends the following as the detailed method to be employed for the next year:

METHOD OF DETERMINING PHOSPHORIC ACID IN FERTILIZERS.

(1) *Prepartion of sample.*—The sample should be well intermixed and properly prepared, so that separate portions shall accurately represent the substance under examination without loss or gain of moisture.

(2) *Determination of moisture.*—(*a*) In potash salts, nitrate of soda, and sulphate of ammonia, heat 1 to 5 grams at 130° C, till the weight is constant, and reckon water from the loss. (*b*) In all other fertilizers, heat 2 grams, or if the sample is too coarse to secure uniform lots of 2 grams each, 5 grams for 5 hours at 100° in a steam bath.

(3) *Water-soluble phosphoric acid.*—Bring 2 grams on a filter; add a little water; let it run out before adding more water, and repeat this treatment cautiously until no phosphate is likely to precipitate in the filter. If the washings show turbidity after passing the filter, clear up with acid. When the substance is nearly washed in this manner, it is transferred to a mortar and rubbed with a rubber-tipped pestle to a homogeneous paste (but not further pulverized), then returned to the filter and washed with water until the filtrate measures not less that 250 cubic centimeters; mix the washings. Take an aliquot (usually corresponding to $\frac{1}{2}$ or $\frac{1}{5}$ gram of the substance), and determine phosphoric acid as under total phosphoric acid.

(4) *Citrate-insoluble phosphoric acid.*—Wash the residue of the treatment with water, into a 150 cubic centimeter flask with 100 cubic centimeters of strictly neutral ammonium citrate solution of 1.09 density; shred and add the filter paper; cork the flask securely; place in a water bath with constant temperature of 65°C., and digest for thirty minutes at this temperature with frequent shaking. Filter the warm solution quickly, and wash with water of ordinary temperature.

Transfer the filter and its contents to a capsule. Ignite until the organic matter is destroyed; treat with 10–15 cubic centimeters of concentrated hydrochloric acid; digest over a low flame until the phosphate is dissolved; dilute to 200 cubic centimeters; mix; pass through a dry filter; take an aliquot and determine phosphoric acid as under total.

In case a determination of citrate insoluble phosphoric acid is required in non-acidulated goods, it is to be made by treating 2 grams of the phosphatic material without previous washing with water, precisely in the way above described, except that in case the substance contains much animal matter (bone, fish, &c.) the residue insoluble in ammonium citrate is to be digested with HCl and $KClO_3$ as described below.

(5) *Total phosphoric acid.*—Weigh 2 grams into a flask or beaker; add 30 cubic centimeters concentrated hydrochloric acid; heat and add cautiously and in small quantities at a time, about 0.5 grams finely-powdered potassium chlorate. Digest at a gentle heat until all phosphates are dissolved and all organic matter destroyed; dilute to 200 cubic centimeters; mix; pass through a dry filter; take 50 cubic centimeters of filtrate; neutralize with ammonia; add about 15 grams dry ammonium nitrate, and to the hot solution, for every decigram of P_2O_5 that is present, 50 cubic centimetres of molybdic solution. Digest at about 65° C. for one hour; filter and wash with ammonium nitrate solution. (Test the filtrate by renewed digestion and addition of more molybdic solution.)

Dissolve the precipitate on the filter with ammonia and hot water, and wash into a beaker to a bulk of not more than 100 cubic centimeters. Nearly neutralize with hydrochloric acid; cool, and add magnesia mixture from a burette, slowly (one drop per second), stirring vigorously. After 15 minutes add 30 cubic centimeters of ammonia solution of density 0.95. Let stand several hours. (Two hours is usually enough.) Filter; wash with dilute ammonia; ignite intensely for 10 minutes and weigh.

(5) *Citrate-soluble phosphoric acid.*—The sum of the water-soluble and citrate insoluble, subtracted from the total gives the citrate-soluble.

PREPARATION OF REAGENTS.

(1) *To prepare ammonium citrate solution.*—Mix 370 grams of commercial citric acid with 1,500 cubic centimeters of water; nearly neutralize with crushed commercial carbonate of ammonia; heat to expel the carbonic acid; cool; add ammonia until exactly neutral (testing by saturated alcoholic solution of coraline) and bring to volume of two liters. Test the gravity (which should be 1.09 at 20°) before using.

(2) *To prepare molybdic solution.*—Dissolve 100 grams of molybdic acid in 400 grams, or 417 cubic centimeters, of ammonic of specific gravity 0.96, and pour the solution thus obtained into 1,500 grams, or 1,250 cubic centimeters, of nitric acid of specific gravity 1.20. Keep the mixture in a warm place for several days, or until a portion heated to 40°C. deposits no yellow precipitate of ammonium phospho-molybdate. Decant the solution from any sediment and preserve in glass-stoppered vessels.

(3) *To prepare ammonium nitrate solution.*—Dissolve 200 grams of commercial ammonium nitrate in water and bring to a volume of 2 liters.

(4) *To prepare magnesia mixture.*—Dissolve 22 grams of recently ignited calcined magnesia in dilute hydrochloric acid, avoiding excess of the latter. Add a little calcined magnesia in excess and boil a few minutes to precipitate iron, alumina, and phosphoric acid; filter; add 280 grams of ammonium chloride, 700 cubic centimeters of ammonia of specific gravity 0.96, and water enough to make the volume of 2 liters. Instead of the solution of 22 grams of calcined magnesia, 110 grams of crystallized magnesium chloride ($MgCl_2.6H_2O$) may be used.

(5) *Dilute ammonia for washing.*—One volume ammonia of specific gravity 0.96 mixed with 3 volumes of water, or, usually, 1 volume of concentrated ammonia with 6 volumes of water.

Respectfully submitted.

H. C. WHITE, *Chairman*,
E. H. JENKINS,
W. C. STUBBS.

On motion of Professor LUPTON this was made the official method for the ensuing year.

Dr. GASCOYNE, from the Committee on Potash, then recommended the following method for determining potash:

METHOD FOR DETERMINING POTASH.

In case the potash is contained in organic compounds like tobacco stems, cotton-seed hulls, &c., it is to be saturated with strong sulphuric acid and ignited in a muffle to destroy organic matter. Pulverize the fertilizer (200 or 300 grams) in a mortar; take 10 grams, boil for ten minutes with 200 cubic centimeters water, and after cooling and without filtering, make up to 1000 cubic centimeters and filter through a dry paper. If the sample have 10–15 per cent. K_2O (kainite), take 50 cubic centimeters of the filtrate; if from 2–3 per cent. K_2O (ordinary potash fertilizers), take 100 cubic centimeters of the filtrate. In each case make the volume up to 150 cubic centimeters, heat to 100°, and add, drop by drop, with constant stirring, slight excess of barium chloride; without filtering, in the same manner add barium hydrate in slight excess. Heat, filter, and wash until precipitate is free of chlorides. Add to filtrate 1 cubic centimeter strong ammonium hydrate, and then a saturated solution of ammonium carbonate until excess of barium is precipitated. Heat. Add now in fine powder 0.5 gram pure oxalic acid or 0.75 gram ammonium oxalate. Filter, wash free of chlorides, evaporate filtrate to dryness in a platinum dish, and, holding dish with crucible tongs, ignite carefully over the free flame below red heat until all volatile matter is driven off.

The residue is now digested with hot water, filtered through a small filter, and washed with successive small portions of water until the filtrate amounts to 30 cubic centimeter or more. To this filtrate, after adding 2 drops of strong hydrochloric acid, is added, in a porcelain dish, 5 to 10 cubic centimeters of a solution of 10 grams of platinic chloride in 100 cubic centimeters of water. The mixture is now evaporated on the water bath to a thick sirup, or further, treated with strong alcohol, washed by decantation, collected in a Gooch crucible or other form of filter, washed with strong alcohol, afterwards with 5 cubic centimeters of ether, dried for 30 minutes at 100° and weighed.

It is recommended that if there is an appearance of white foreign matter in the double salt it should be washed with 10 cubic centimeters of a half concentrated solution of NH_4Cl which has been saturated by shaking with K_2PtCl_6, rejecting the first portion running through and using 10cc. of fresh solution, returning the latter portion five or six times upon the salt and finally using another portion, and then alcohol and ether, after the method recommended by Gladding.

As an alternative the entire method of Lindo, as modified by Gladding, may be used

in the determination of potash when, in the opinion of the analyst, it is found satisfactory.

The use of the factor 0.33056 for converting K_2PtCl_6 to KCl and .19308 for converting to K_2O are continued.

Respectfully submitted.

W. J. GASCOYNE, *Chairman.*
CLIFFORD RICHARDSON.
M. A. SCOVELL.

On motion of Dr. WHITE the recommendations were adopted as the official method for the ensuing year.

Dr. GASCOYNE then presented the report of the Committee on Nitrogen, embracing recommendations for the use of the Ruffle method.

Professor STUBBS desired to know if, were this report adopted, the Ruffle method alone would be official, and this being apparently the case, he dissented from its adoption.

Dr. JENKINS said that he felt that he could not give up the Kjeldahl and absolute method, but that were the Ruffle adopted as official he should check his results by that.

Considerable opposition to the adoption of any one method being apparent, it was moved and passed—

That no single method be selected as official, but that the Association recognize as such either the Ruffle, Kjeldahl, absolute, or soda-lime methods, when carried out according to working details to be supplied by the committee.

These details are presented in the Appendix.

Miscellaneous business being in order, the Committee on a Uniform Law presented the following report:

The committee appointed at the last annual session "to consider the question of uniformity of legislation and regulations as to the inspection of fertilizers, and to prepare a draft of a uniform law," and to whom the communication of the National Fertilizer Association presented through Mr. de Ghequier has been referred, has taken these matters under consideration and begs leave to report:

(1) The committee is of opinion that no recommendation concerning national legislation should be made by this Association.

(2) The committee recommends that the communications presented by Mr. de Ghequier be spread on the minutes.

(3) The conditions in the different States are so various that it is clearly impracticable to draft a uniform law covering the details of inspection, regulation of sale, &c., of commercial fertilizers that would be generally acceptable.

(4) With the legal and commercial features of such legislation this Association has nothing to do. The form of statement of the analysis of fertilizers is a matter, however, the discussion of which is clearly within our province. The committee suggests the following as a proper form in which guaranteed and official analyses should be expressed:

	Per cent.
Moisture	
Soluble phosphoric acid	
Reverted phosphoric acid	
Insoluble phosphoric acid	
Total available phosphoric acid	
Total phosphoric acid	
Nitrogen	
Equivalent to ammonia	
Potash (K_2O)	

The committee further suggests that the same form, or such part or parts of it as may be necessary, be recommended as proper to be attached to bags or packages containing fertilizers.

And the committee recommends that each member of this Association use his influence to have a clause inserted in the fertilizer law of his State looking to the adoption of the forms of analysis above expressed.

Respectfully submitted.

H. C. WHITE, *Chairman.*
P. E. CHAZAL.
E. H. JENKINS.

Professor STUBBS moved to strike out the words "total available" in the form recommended by the committee, which was carried, and the report was then adopted.

The Committee on Nominations then reported the following list:

For President: Dr. E. H. Jenkins, of the Connecticut Agricultural Experiment Station.

For Vice-President: Mr. P. E. Chazal, State chemist of South Carolina.

For Secretary and Treasurer: Clifford Richardson, of the United States Department of Agriculture.

As Members of the Executive Committee: Dr. H. W. Wiley, of the United States Department of Agriculture; Prof. M. A. Scovell, of the Kentucky Agricultural Experiment Station.

The report of the committee was adopted, and by unanimous consent the secretary was instructed to cast the vote of the Association for the candidates named, and they were declared elected.

Professor LUPTON then moved, and it was carried, that the place of meeting next year be Washington.

Professor LUPTON then moved, and it was carried, that the executive committee inquire into the advisability of incorporating the Association, to report at the next meeting.

Dr. JENKINS moved, and Mr. CHAZAL seconded, the following resolution, which was carried unanimously:

Resolved, That this Association hereby tenders to the honorable Commissioner of Agriculture its hearty thanks for the use of the rooms in which its meetings have been held; for the numerous courtesies which he and the members of his Department have extended to it during its present session, and also for his generous offer of issuing the results of its deliberations as a special bulletin of the Department of Agriculture. We believe that this action has been already recognized as a substantial aid to all interested either in the manufacture or practical use of commercial fertilizers.

The thanks of the Association to the president and secretary were then moved and carried, and, on motion of Dr. White, the convention adjourned *sine die.*

Appendix A.

OFFICIAL METHODS OF THE ASSOCIATION OF AGRICULTURAL CHEMISTS FOR 1886-'87.

METHOD FOR DETERMINING PHOSPHORIC ACID AND MOISTURE.

(1) *Preparation of sample.*—The sample should be well intermixed and properly prepared, so that separate portions shall accurately represent the substance under examination, without loss or gain of moisture.

(2) *Determination of moisture.*—(*a*) In potash salts, nitrate of soda, and sulphate of ammonia heat 1 to 5 grams at 130° C. till the weight is constant, and reckon water from the loss. (*b*) In all other fertilizers heat 2 grams, or if the sample is too coarse to secure uniform lots of 2 grams each, 5 grams for five hours at 100° in a steam bath.

(3) *Water-soluble phosphoric acid.*—Bring 2 grams on a filter, add a little water, let it run out before adding more water, and repeat this treatment cautiously until no phosphate is likely to precipitate in the filter. If the washings show turbidity after passing the filter clear up with acid. When the substance is nearly washed in this manner it is transferred to a mortar and rubbed with a rubber tipped pestle to a homogeneous paste (but not further pulverized), then returned to the filter and washed with water until the filtrate measures not less than 250 cubic centimeters. Mix the washings. Take an aliquot (usually corresponding to $\frac{1}{5}$ or $\frac{1}{4}$ gram of the substance) and determine phosphoric acid, as under total phosphoric acid.

(4) *Citrate-insoluble phosphoric acid.*—Wash the residue of the treatment with water into a 150 cubic centimeter flask with 100 cubic centimeters of strictly neutral ammonium citrate solution of 1.09 density, shred and add the filter paper, cork the flask securely, place in a water bath with constant temperature of 65° C., and digested for thirty minutes at this temperature with frequent shaking, filter the warm solution quickly and wash with water of ordinary temperature.* Transfer the filter and its contents to a capsule, ignite until the organic matter is destroyed, treat with 10 to 15 cubic centimeters of concentrated hydrochloric acid, digest over a low flame until the phosphate is dissolved, dilute to 200 cubic centimeters, mix, pass through a dry filter, take an aliquot and determine phosphoric acid as under total.

In case a determination of citrate-insoluble phosphoric acid is required in non-acidulated goods, it is to be made by treating 2 grams of the phosphatic material without previous washing with water, precisely in the way above described, except that in case the substance contains much animal matter (bone, fish, &c.) the residue insoluble in ammonium citrate is to be digested with HCl and $KClO_3$, as described below.

(5) *Total phosphoric acid.*—Weigh 2 grams into a flask or beaker, add 30 cubic centimeters concentrated hydrochloric acid, heat and add cautiously and in small quantities at a time about 0.5 gram finely-powdered potassium chlorate. Digest at a gentle heat until all phosphates are dissolved and all organic matter destroyed, dilute to 200 cubic centimeters; mix; pass through a dry filter: take 50 cubic centimeters of filtrate; neutralize with ammonia; for every decigram of P_2O_5 that is pres-

e
nt. 50 cubic centimeters of molybdic solution. Digest at about 65° C. for one hour filter and wash with ammonium nitrate solution. (Test the filtrate by renewed digestion and addition of more molybdic solution.) Dissolve the precipitate on the filter with ammonia and hot water, and wash into a beaker to a bulk of not more than 100 cubic centimeters. Nearly neutralize with hydrochloric acid, cool, and add magnesia mixture from a burette; add slowly (one drop per second), stirring vigorously. After 15 minutes add 30 cubic centimeters of ammonia solution of density 0.95. Let stand several hours (two hours is usually enough). Filter; wash with dilute ammonia; ignite intensely for 10 minutes and weigh.

(6) Citrate-soluble phosphoric acid. The sum of the water-soluble and citrate-insoluble subtracted from the total gives the citrate-soluble.

PREPARATION OF REAGENTS.

(1) *To prepare ammonium citrate solution.*—Mix 370 grams of commercial citric acid with 1,500 cubic centimeters of water; nearly neutralize with crushed commercial carbonate of ammonia; heat to expel the carbonic acid; cool; add ammonia until exactly neutral (testing by saturated alcoholic solution of coralline) and bring to volume of two liters. Test the gravity, which should be 1.09 at 20°, before using.

(2) *To prepare molybdic solution.*—Dissolve 100 grams of molybdic acid in 400 grams or 417 cubic centimeters of ammonic of specific gravity 0.96, and pour the solution thus obtained into 1,500 grams or 1,250 cubic centimeters of nitric acid of specific gravity 1.20. Keep the mixture in a warm place for several days, or until a portion heated to 40° C. deposits no yellow precipitate of ammonium phospho-molybdate. Decant the solution from any sediment, and preserve in glass-stoppered vessels.

(3) *To prepare ammonium nitrate solution.*—Dissolve 200 grams of commercial ammonium nitrate in water and bring to a volume of two liters.

(4) *To prepare magnesia mixture.*—Dissolve 22 grams of recently-ignited calcined magnesia in dilute hydrochloric acid, avoiding excess of the latter. Add a little calcined magnesia in excess, and boil a few minutes to precipitate iron, alumina, and phosphoric acid, filter, add 280 grams of ammonium chloride, 700 cubic centimeters of ammonia of specific gravity 0.96, and water enough to make the volume of two liters. Instead of the solution of 22 grams of calcined magnesia 110 grams of crystallized magnesium chloride ($MgCl_2$, $6H_2O$) may be used.

(5) *Dilute ammonia for washing.*—One volume ammonia of specific gravity 0.96 mixed with three volumes of water, or usually 1 volume of concentrated ammonia with 6 volumes of water.

METHODS FOR DETERMINING POTASH.

In case the potash is contained in organic compounds like tobacco stems, cotton-seed hulls, &c., it is to be saturated with strong sulphuric acid and ignited in a muffle to destroy organic matter. Pulverize the fertilizer (200 or 300 grams) in a mortar; take 10 grams, boil for ten minutes with 200 cubic centimeters water, and after cooling, and without filtering, make up to 1,000 cubic centimeters, and filter through a dry paper. If the sample have 10 to 15 per cent. K_2O (kainite), take 50 cubic centimeters of the filtrate: if from 2 to 3 per cent. K_2O (ordinary potash fertilizers), take 100 cubic centimeters of the filtrate. In each case make the volume up to 150 cubic centimeters, heat to 100°, and add, drop by drop, with constant stirring, slight excess of barium chloride; without filtering, in the same manner, add barium hydrate in slight excess. Heat, filter, and wash until precipitate is free of chlorides. Add to filtrate 1 cubic centimeter strong ammonium hydrate, and then a saturated solution of ammonium carbonate until excess of barium is precipitated. Heat. Add now, in fine powder, 0.5 gram pure oxalic acid or 0.75 gram ammonium oxalate. Filter, wash free of chlorides, evaporate filtrate to dryness in a platinum dish, and, holding dish with crucible tongs, ignite carefully over the free flame below red heat until all volatile matter is driven off.

The residue is now digested with hot water, filtered through a small filter, and washed with successive small portions of water until the filtrate amounts to 30 cubic centimeters or more. To this filtrate, after adding two drops of strong hydrochloric acid, is added, in a porcelain dish, 5 to 10 cubic centimeters of a solution of 10 grams of platinic chloride in 100 cubic centimeters of water. The mixture is now evaporated on the water-bath to a thick sirup or further, treated with strong alcohol washed by decantation, collected in a Gooch crucible or other form of filter, washed with strong alcohol, afterwards with 5 cubic centimeters ether, dried for thirty minutes at 100°, and weighed.

It is recommended that if there is an appearance of white foreign matter in the double salt it should be washed with 10 cubic centimeters of a half-concentrated solution of NH_4Cl, which has been saturated by shaking with K_2PtCl_6, rejecting the first portion running through and using 10 cubic centimeters of fresh solution, returning the same portion five or six times upon the salt, and finally using another portion, and then alcohol and ether, after the method recommended by Gladding.

As an alternative the entire method of Lindo, as modified by Gladding, may be used in the determination of potash when, in the opinion of the analyst, it is found satisfactory.

The use of the factor 0.3056 for converting K_2PtCl_6 to KCl and 0.19308 for converting to K_2O are continued.

METHOD OF LINDO AS MODIFIED BY GLADDING.

(1) *Superphosphates.*—Boil 10 grams of the fertilizer with 300 cubic centimeters of water for ten minutes. Cool the solution, add ammonia in slight excess, thus precipitating all phosphate of lime, oxide of iron, and alumina, &c., make up to 500 cubic centimeters, mix thoroughly, and filter through a dry filter; take 50 cubic centimeters, corresponding to 1 gram, evaporate nearly to dryness, add 1 cubic centimeter of dilute H_2SO_4 (1 to 1), and evaporate to dryness and ignite to whiteness. As all the potash is in form of sulphate, no loss need be apprehended by volatilization of potash, and a full red heat must be used until the residue is perfectly white. This residue is dissolved in hot water plus a few drops of HCl; 5 cubic centimeters of a solution of pure NaCl (containing 20 grams NaCl to the liter) and an excess of platinum solution (4 cubic centimeters) are now added, and the whole evaporated as usual. The precipitate is washed thoroughly with alcohol by decantation and on filter, as usual. The washing should be continued even after the filtrate is colorless. 10 cubic centimeters of the NH_4Cl solution prepared as above are now run through the filter. These 10 cubic centimeters will contain the bulk of the impurities, and are thrown away. A fresh portion of 10 cubic centimeters NH_4Cl is now run through the filter several times (5 or 6). The filter is then washed thoroughly with pure alcohol, dried, and weighed as usual. The platinum solution used contains 1 gram metallic platinum in every 10 cubic centimeters.

(2) *Muriates of potash.*—In the analysis of these salts an aliquot portion, containing .500 gram, is evaporated with 10 cubic centimeters platinum solution plus a few drops of HCl, and washed as before.

(3) *Sulphate of potash, kainite, &c.*—In the ananlysis of these salts an aliquot portion containing .500 gram is taken, .250 gram of NaCl added, plus a few drops of HCl, and the whole evaporated with 15 cubic centimeters platinum solution. In this case special care must be taken, in the washing with alcohol, to remove all the double chloride of platinum and sodium. The washing should be continued for some time after the filtrate is colorless. 25 cubic centimeters of the NH_4Cl solution are employed, instead of 10 cubic centimeters, and the second 25 cubic centimeters poured through at least six times to remove all sulphates and chlorides. Wash finally with alcohol, dry, and weigh as usual.

To prepare the washing solution of NH_4Cl, place in a bottle 500 cubic centimeters H_2O, 100 grams of NH_4Cl; shake till dissolved. Now pulverize 5 or 10 grams of K_2PtCl_6, put in the bottle, and shake at intervals for six or eight hours; let settle over night; then filter off liquid into a second bottle. The first bottle is then ready for a preparation of a fresh supply when needed.

DETERMINATION OF NITROGEN BY THE ABSOLUTE OR CUPRIC OXIDE METHOD.

The apparatus and reagents needed are as follows:

APPARATUS.

Combustion tube of best hard Bohemian glass, about 26 inches long and one-half inch internal diameter.

Azotometer of at least 100 cubic centimeters capacity accurately calibrated.

Sprengel mercury air pump.

Small paper scoop, easily made from stiff writing-paper.

REAGENTS.

Cupric oxide (coarse).—Wire form; to be ignited and cooled before using.

Fine cupric oxide.—Prepared by pounding ordinary cupric oxide in mortar.

Metallic copper.—Granulated copper or fine copper gauze reduced and cooled in stream of hydrogen.

Sodium bicarbonate.—Free from organic matter.

Caustic potash solution.—Dissolve commercial stick potash in less than its weight of water so that crystals are deposited on cooling. When absorption of carbonic acid ceases to be prompt solution must be discarded.

LOADING TUBE.

Of ordinary commercial fertilizers take 1 to 2 grams for analysis. In the case of highly nitrogenous substances the amount to be taken must be regulated by the amount of nitrogen estimated to be present. Fill tube as follows: (1) About 2 inches of coarse cupric oxide. (2) Place on the small paper scoop enough of the fine cupric oxide to fill, after having been mixed with the substance to be analyzed, about 4 inches of the tube; pour on this the substance, rinsing watch glass with a little of the fine oxide and mix thoroughly with spatula; pour into tube, rinsing the scoop with a little fine oxide. (3) About 12 inches of coarse cupric oxide. (4) About 3 inches of metallic copper. (5) About 2½ inches of coarse cupric oxide (anterior layer). (6) Small plug of asbestos. (7) Eight-tenths to 1 gram of sodium bicarbonate. (8) Large, loose plug of asbestos; place tube in furnace, leaving about 1 inch of it projecting; connect with pump by rubber stopper smeared with glycerine, taking care to make connection perfectly tight.

OPERATION.

Exhaust air from tube by means of pump. When a vacuum has been obtained, allow flow of mercury to continue, light gas under that part of tube containing metallic copper, anterior layer of cupric oxide (see 5th above) and bicarbonate of soda. As soon as vacuum is destroyed and apparatus filled with carbonic acid gas, shut off the flow of mercury and at once introduce the delivery tube of the pump into the receiving arm of the azotometer and just below the surface of the mercury seal of the azotometer, so that the escaping bubbles will pass into the air and not into the azotometer, thus avoiding the useless saturation of the caustic potash solution.

When the flow of carbonic acid has very nearly or completely ceased, pass the delivery tube down into the receiving arm so that the bubbles will escape into the azo-

tometer. Light the jets under the 12-inch layer of oxide, heat gently for a few moments to drive out any moisture that may be present and bring to red heat. Heat gradually mixture of substance and oxide, lighting one jet at a time. Avoid too rapid evolution of bubbles, which should be allowed to escape at rate of about one per second or a little faster.

When the jets under mixture have all been turned on, light jets under layer of oxide at end of tube. When evolution of gas has ceased, turn out all the lights except those under the metallic copper and anterior layer of oxide, and allow to cool for a few moments. Exhaust with pump and remove azotometer before flow of mercury is stopped. Break connection of tube with pump, stop flow of mercury and extinguish lights. Allow azotometer to stand for at least an hour or cool with stream of water until permanent volume and temperature are reached.

Adjust accurately the level of the KOH solution in bulb to that in azotometer, note volume of gas, temperature, and height of barometer; make calculations as usual. The labor of calculation may be much diminished by the use of the tables prepared by Messrs. Battle and Dancy, of the North Carolina Experiment Station (Raleigh, N. C.).

The above details are, with some modifications, those given in the report of the Connecticut Station for 1879 (p. 124), which may be consulted for details of apparatus, should such details be desired.

DETERMINATION OF NITROGEN BY THE METHOD OF KJELDAHL.

REAGENTS AND APPARATUS.

(1) Hydrochloric acid whose absolute strength has been determined, (a) by precipitating with silver nitrate and weighing the silver chloride, (b) by sodium carbonate, as described in Fresenius's Quantitative Analysis, second American edition, page 680, and (c) by determining the amount neutralized by the distillate from a weighed quantity of pure ammonium chloride boiled with an excess of sodium hydrate.

(2) Standard ammonia whose strength, relative to the acid, has been accurately determined.

(3) "C. P." sulphuric acid, Sp. Gr. 1.83, free from nitrates and also from ammonium sulphate, which is sometimes added in the process of manufacture to destroy oxides of nitrogen.

(4) Mercuric oxide, HgO, prepared in the wet way. That prepared from mercury nitrate cannot safely be used.

(5) Potassium permanganate tolerably finely pulverized.

(6) Granulated zinc.

(7) A solution of 40 grams of commercial potassium sulphide in one liter of water.

(8) A saturated solution of sodium hydrate free from nitrates, which are sometimes added in the process of manufacture to destroy organic matter and improve the color of the product.

(9) Solution of cochineal prepared according to Fresenius's Quantitative Analysis, second American edition, page 679.

(10) Burettes should be calibrated in all cases by the user.

(11) Digestion flasks of hard, moderately thick, well-annealed glass. These flasks are about 9 inches long, with a round, pear-shaped bottom, having a maximum diameter of 2½ inches, and tapering out gradually in a long neck, which is three-fourths of an inch in diameter at the narrowest part, and flared a little at the edge. The total capacity is 225 to 250 cubic centimeters.

(12) Distillation flasks of ordinary shape, 550 cubic centimeters' capacity, and fitted with a rubber stopper and a bulb tube above to prevent the possibility of sodium hydrate being carried over mechanically during distillation. This is adjusted to the tube of the condenser by a rubber tube.

(13) A condenser. Several forms have been described, no one of which is equally

convenient for all laboratories. The essential thing is that the tube which carries the steam to be condensed shall be of block tin. All kinds of glass are decomposed by steam and ammonia vapor, and will give up alkali enough to impair accuracy. (See Kreussler and Henzold, Ber. Berichte, *XVII*, 34.) The condenser in use in the laboratory of the Conn. Exp. Station, devised by Professor Johnson, consists of a copper tank supported by a wooden frame, so that its bottom is 11 inches above the work-bench on which it stands. This tank is 16 inches high, 32 inches long, and 3 inches wide from front to back, widening above to 6 inches. It is provided with a water-supply tube which goes to the bottom and a larger overflow pipe above. The block tin condensing tubes, whose external diameter is $\frac{5}{8}$ of an inch, 7 in number, enter the tank through holes in the front side of it near the top, above the level of the overflow, and pass down perpendicularly through the tank and out through rubber stoppers tightly fitted into holes in the bottom. They project about $1\frac{1}{2}$ inches below the bottom of the tank, and are connected by short rubber tubes with glass bulb tubes of the usual shape, which dip into glass precipitating beakers. These beakers are $6\frac{1}{2}$ inches high, 3 inches in diameter below, somewhat narrower above, and of about 500 cubic centimeters capacity. The titration can be made directly in them. The seven distillation flasks are supported on a sheet-iron shelf attached to the wooden frame that supports the tank in front of the latter. Where each flask is to stand a circular hole is cut, with three projecting lips, which support the wire gauze under the flask, and three other lips which hold the flask in place and prevent its moving laterally out of place while distillation is going on. Below this sheet-iron shelf is a metal tube carrying seven Bunsen burners, each with a stop-cock like those of a gas combustion furnace. These burners are of larger diameter at the top, which prevents smoking when covered with fine gauze to prevent the flame from striking back.

(14) The stand for holding the digestion flasks consists of a pan of sheet-iron 29 inches long by 8 inches wide, on the front of which is fastened a shelf of sheet-iron as long as the pan, 5 inches wide and 4 inches high. In this are cut six holes $1\frac{5}{8}$ inches in diameter. At the back of the pan is a stout wire running lengthwise of the stand, 8 inches high, with a bend or depression opposite each hole in the shelf. The digestion flask rests with its lower part over a hole in the shelf and its neck in one of the depressions in the wire frame, which holds it securely in position. Heat is supplied by low Bunsen burners below the shelf. Dr. Jenkins has used asbestus paper under the flasks, but finds that with a little care the naked flame can be applied directly to the flask without danger.

THE DETERMINATION.

One gram of the substance to be analyzed is brought into a digestion flask with approximately 0.7 gram of mercuric oxide and 20 cubic centimeters of sulphuric acid. The flask is placed on the frame above described in an inclined position and heated below the boiling point of the acid for from 5 to 15 minutes, or until frothing has ceased. The heat is then raised till the acid boils briskly. No further attention is required till the contents of the flask has become a clear liquid, which is colorless or at least has only a very pale straw color. The flask is then removed from the frame, held upright, and while still hot, potassium permanganate is dropped in carefully and in small quantity at a time till after shaking the liquid remains of a green or purple color. After cooling, the contents of the flask are transferred to the distilling flask with water, and to this 25 cubic centimeters of potassium sulphide solution are added, 50 cubic centimeters of the soda solution, or sufficient to make the reaction strongly alkaline, and a few pieces of granulated zinc. The flask is at once connected with the condenser and the contents of the flask are distilled till all ammonia has passed over into the standard acid contained in the precipitating flask previously described and the concentrated solution can no longer be safely boiled. This operation usually requires from 20 to 40 minutes. The distillate is then titrated with standard ammonia.

The use of mercuric oxide in this operation greatly shortens the time necessary for

digestion, which is rarely over an hour and a half in the case of substances most difficult to oxidize and is more commonly less than an hour. In most cases the use of potassium permanganate is quite unnecessary, but it is believed that in exceptional cases it is required for complete oxidation, and in view of the uncertainty it is always used. Potassium sulphide removes all mercury from solution and so prevents the formation of mercuro-ammonium compounds which are not completely decomposed by soda solution. The addition of zinc gives rise to an evolution of hydrogen and prevents violent bumping. Previous to use the reagents should be tested by a blank experiment with sugar, which will partially reduce any nitrates that are present which might otherwise escape notice.

This method cannot be used for the determination of nitrogen in substances which contain nitrates or certain albuminoids.

The following references to papers in which this method is discussed may be of service:

Kjeldahl's original paper, Fres. Zeitschr., 1883, 22, p. 366.

Descriptions of apparatus, modifications of the process, precautions to be observed, control determinations, &c.

Maercker, Fres. Zeitschr. 1884, 223, 559. Chem. Zeitg., *VIII*, 432. Stutzer und Reitmar, Chem. Zeitg., 1885, No. 76, from Rep. anal. Chem., 1885, *V*, 232. Pfeiffer und Lehmann, Fres. Zeitschr., 1885, *XXIV*, 388. Kreussler, Fres. Zeitschr., 1885, *XXIV*, 393. Kreussler, Fres. Zeitschr., 1885, *XXIV*, 453. Landw. Versuchs, Stationen, *XXXI*, 269. Dafert, Fres. Zeitschr., 1885, *XXIV*, 454. Wilfarth, Fres. Zeitschr., 1885, *XXIV*, 455. Chem. Centralblatt (3 F.), *XVI*, 17 and 113. Rindell und Hannin, Fres. Zeitschr. 1886, *XXV*, 155. Czeczetka, Fres. Zeitschr., 1886, *XXV*, 252.

Application to determination of nitrogen in *coke and coal*. Sigismund und Schmitz, Fres. Zeitschr., 1886, *XXV*, 314. In *urine*, Petri und Lehmann, Zeitschr. für physiolog. Chem., *VIII*, 200. Fres. Zeitschr. 1884, *XXIII*, 596. Pfluger und Bohland, Pfluger's Archiv *XXXV*, 454. Fres. Zeitschr., 1885, *XXIV*, 299. Arnold, Répertoire d. anal. Chem., *IV*, 97. Hofmeister, Fres. Zeitschr., *XXIV*, 635. Bohland, Pfluger's Archiv *XXXVII*, 423. Fres. Zeitschr., 1886, *XXV*, 280. In *wine, must*, and *yeast*, Fres. Zeitschr., 1886, *XXV*, 149. In *asparagin, aspartic acid, glutamin, glutamic acid, leucin, tyrosin, allantoin*, &c. Bosshard, Fres. Zeitschr., 1885, *XXIV*, 199.

Application to physiological experiments in general. Arnold, Archiv d. Phar. (3 R.), *XXIII*, 177. Fres. Zeitschr., 1886, *XXV*, 454.

DETERMINATION OF NITROGEN BY THE RUFFLE METHOD.

PREPARATION OF REAGENTS.

(1) *A standard solution of sulphuric acid*, half-normal, or 19.968 grams SO_3 per liter.

(2) *A standard solution of potassium hydrate*, half-normal, or 27.991 grams KOH per liter.

(3) *An alcoholic solution of coralline.*

(4) *Hyposulphite mixture.*—Prepared by mixing equal parts by weight of dry slaked lime and finely-powdered crystallized sodium hyposulphite. This may be made in quantity, as it will keep dry for any length of time if kept in a well-stoppered bottle.

(5) *Charcoal and sulphur mixture.*—This is prepared by mixing equal parts by weight of finely-powdered wood charcoal (N. free) and flowers of sulphur. This must be kept in a glass-stoppered bottle. The purity of the reagents must be tested by a blank combustion.

(6) *Ordinary granulated soda-lime.*

APPARATUS.

(1) *Combustion tubes* of hard Bohemian glass, 20 inches long and $\frac{1}{2}$ inch internal diameter, drawn to a point.

(2) *Three bulb, 6-inch U tubes*, with glass stop-cock.

(3) *Aspirator.*

PREPARATION.

(1) Clean and fill the U tube with 10 cubic centimeters of standard acid.

(2) Fit cork and glass connecting tube. Fill the tube as follows: (1) A loosely fitting plug of asbestus, previously ignited, and then 1 to 1½ inches of the hyposulphite mixture. (2) The weighed portion of the substance to be analyzed is intimately mixed with an equal weight of the charcoal and sulphur mixture. (3) Pour on a piece of glazed paper, or porcelain mortar, a sufficient quantity of the hyposulphite mixture to fill about 10 inches of the tube; then add the substance to be analyzed, as previously prepared; mix carefully and pour into the tube; shake down the contents of the tube; rinse off the paper or mortar with a small quantity of the hyposulphite mixture and pour into the tube; then fill up with soda-lime to within 2 inches of the end of the tube. (4) Place another plug of ignited asbestus at the end of the tube, and close with a cork. (5) Hold the tube in a horizontal position, and tap on the table until there is a gas channel all along the top of the tube. Place in the furnace and make connection with the U tube containing the acid. Aspirate and see that the apparatus is tight.

The combustion.—Place the prepared combustion tube in the furnace, letting the open end project a little so as not to burn the cork. Commence by heating the soda-lime portion until it is brought to a full red heat. Then turn up slowly jet after jet toward the other end of the tube, so that the bubbles come off two or three a second. When the whole tube is red hot and the evolution of the gas has ceased and the liquid in the U tube begins to recede towards the furnace, attach the aspirator to the other limb of the U tube, break off the end of the tube, and draw a current of air through for a few minutes. Detach the U tube and wash the contents into a breaker or porcelain basin, add a few drops of the coralline solution and titrate.*

DETERMINATION OF NITROGEN BY THE SODA-LIME METHOD.

Select a tube of hard glass 14 to 16 inches long, draw one end of it to a fine point, and to the other end fit a cork, through which is passed a tube bent at right angles, the other end of which passes through a cork closing tightly one arm of a 6-inch three-bulbed U tube with glass stop-cock. Into the combustion tube first slip a loosely fitting plug of asbestus previously ignited, and then 1½ to 2 inches of soda-lime. Weigh out from 0.7 to 2.8 grams of the substance to be analyzed, and mix it on a piece of glazed paper or porcelain mortar with some finely pulverized soda-lime, and introduce the mixture into the combustion-tube. The paper or mortar is then rinsed out with a small quantity of soda-lime and poured into the tube. The tube is then filled up to within 1 to 2 inches of the open end with granulated soda-lime, then fill up with a plug of ignited asbestus. A free passage is formed for the evolved gases by holding the tube in a horizontal position and tapping gently on the table. Introduce the prepared combustion tube into the furnace, letting the open end project a little so as not to burn the cork, supporting the U tube by a clamp. The tube is then gradually heated, commencing at the fore part, nearest the cork, and progressing slowly towards the tail. The combustion should be conducted so as to obtain a steady and uninterrupted flow of gas. When properly carried out the acid in the U tube is never colored. When the acid begins to recede attach the aspirator to the other limb of the U tube, and start it slowly, then break off the point of the combustion tube and draw a current of air through the apparatus for a few minutes, in order to sweep all the ammonia into the acid.

Detach the U tube and wash the contents into a breaker or porcelain basin, add a few drops of an alcoholic solution of coralline and titrate.

The standard acid, alkali, and indicator to be the same as that used in the Ruffle method.

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*Cochineal may be found a better indicator, as the H_2S evolved in the combustion affects corralline.—(C. R).

Appendix B

COMMITTEES OF THE ASSOCIATION FOR 1886-'7, APPOINTED BY THE PRESIDENT.

PHOSPHORIC ACID.

Prof. W. C. Stubbs, Baton Rouge, La.
Prof. W. E. Moses, Knoxville, Tenn.
Dr. C. W. Dabney, Jr., Raleigh, N. C.

NITROGEN.

Dr. W. J. Gascoyne, Richmond, Va.
Mr. P. E. Chazal, Columbia, S. C.
Prof. M. A. Scovell, Lexington, Ky.

POTASH.

Mr. Clifford Richardson, Washington, D. C.
Prof. H. A. Huston, Lafayette, Ind.
Prof. W. W. Cook, Burlington, Vt.

FEEDING STUFFS.

Dr. G. C. Caldwell, Ithaca, N. Y.
Prof. W. H. Jordan, Orono, Me.
Mr. Clifford Richardson, Washington, D. C.

DAIRY PRODUCTS.

Dr. H. W. Wiley, Washington, D. C.
Dr. S. M. Babcock, Geneva, N. Y.
Prof. H. P. Armsby, Madison, Wis.

Appendix C.

CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This Association shall be known as the Association of Official Agricultural Chemists in the United States. The objects shall be (1) to secure uniformity and accuracy in the methods, results, and modes of statements of analysis of fertilizers, soils, cattle-foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with departments of agriculture, agricultural experiment stations, agricultural colleges, State boards of agriculture, and other bodies charged with official control of the materials named in section 1, shall alone be eligible to membership, and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to a vote in the Association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. Any person eligible to membership may become a member at any meeting of the Association by presenting proper credentials and signing this constitution. All analytical chemists and others interested in the objects of the Association may attend its meetings and take part in its discussions, but shall have no vote in the Association.

(3) The officers of the Association shall consist of a president, vice-president, and secretary, who shall also officiate as treasurer; and these officers, together with two other members to be elected by the Association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board, whose members are eligible to membership, his office shall be considered vacant, and a successor may be appointed by the executive committee to continue in office till the annual meeting next following.

(4) There shall be appointed by the chair, at the regular annual meeting, standing committees of three members each, to be called, respectively, the committee on phosphoric acid, the committee on potash, the committee on nitrogen, the committee on cattle-foods, and the committee on dairy products, whose reports shall be taken up, discussed, and disposed of in the order named. Additional committees may be appointed from time to time as the Association may direct. It shall be the duty of these committees to test methods, to distribute samples to members of the association and others who may signify their desire to participate in the work, and to present a written report at each annual meeting embodying the progress made during the year in analytical methods bearing on their respective topics, and the results of work done under their direction.

(5) The special duties of the officers of the Association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this Association shall be held at such place as shall be decided by the Association, and at such time as shall be decided by the executive committee, and announced at least three months before the time of meeting.

(7) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(8) The executive committee shall confer with the official boards represented with reference to the payment of expenses connected with the meetings and publication of the proceedings of the Association.

(9) All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by a vote of two-thirds of the members present and entitled to vote.

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